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PROCEEDINGS
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American Society for Testing Materials

ORGANIZED IN 1898
INCORPORATED IN 1902

PROCEEDINGS, PART II

SUMMARY OF PROCEEDINGS OF THE CHICAGO REGIONAL MEETING—SYMPOSIUM ON CORROSION TESTING PROCEDURES; SYMPOSIUM ON LUBRICANTS

The eighth regional meeting of the Society was held in Chicago, Ill., at the Palmer House on Tuesday and Wednesday, March 2 and 3, 1937, in conjunction with the Spring Group Meeting of A.S.T.M. Committees. Arrangements for the regional meeting were under the auspices of the Chicago District Committee.

Symposium on Corrosion Testing Procedures:

The Tuesday evening session of the meeting comprised a technical program, a Symposium on Corrosion Testing Procedures sponsored jointly by the Society's Committees A-5 on Corrosion of Iron and Steel, and B-3 on Corrosion of Non-Ferrous Metals and Alloys. The personnel of the joint committee immediately in charge of the technical program (F. N. Speller, chairman) consisted of the following:

Representing Committee A-5:
- F. N. Speller, Director, Department of Metallurgy and Research, National Tube Co.
- L. W. Hopkins, Materials Engineer, American Chain and Cable Co., Inc.

Representing Committee B-3:
- R. J. McKay, Superintendent, Technical Service, The International Nickel Co., Inc.
- C. L. Hippensteel, Member of Technical Staff, Bell Telephone Laboratories, Inc.

The purpose of the symposium was to study the desirable steps to be taken in the direction of standardization of corrosion testing methods. The symposium consisted of the following papers:

"Introduction," by F. N. Speller.
"The Principles of Corrosion Testing," by C. W. Borgmann, Research Engineer, National Tube Co., and R. B. Mears, Metallurgist, Aluminum Research Laboratories, Aluminum Company of America.

"Atmospheric Corrosion Testing," by Henry S. Rawdon, Chief, Division of Metallurgy, National Bureau of Standards.

"Salt-Spray Testing," by E. H. Dix, Jr., Chief Metallurgist, and J. J. Bowman, Metallurgical Division, Aluminum Research Laboratories, Aluminum Company of America.

"An Electrical Resistance Method of Determining Corrosion Rates," by R. R. Seeber, Professor of Mechanical Engineering, Michigan College of Mining and Technology.

"Alternate-Immersion and Water-Line Tests," by D. K. Crampton, Director of Research, and N. W. Mitchell, Research Metallurgist, Chase Brass and Copper Co.

"Standardizing Liquid Corrosion Tests," by R. J. McKay, Chemical Engineer, Nickel Sales Dept., and F. L. LaQue, Development and Research Division, The International Nickel Co., Inc.

"Soil Corrosion Testing," by K. H. Logan, Chief, S. P. Ewing, Research Associate of the American Gas Assn., and I. A. Denison, Chemist, Underground Corrosion Section, National Bureau of Standards, Washington, D. C.

The co-chairmen at the session were Messrs. Speller and McKay.

A considerable amount of discussion resulted from the single session held at the regional meeting. However, the committee in charge felt that for a subject of such importance further opportunity should be given for discussion. A session of the Fortieth Annual Meeting was therefore set aside for this purpose. The discussion from the regional and annual meetings of the Society was carefully reviewed, abstracted, and combined. The papers, complete with this discussion, have been published by the Society as a separate volume entitled, "Symposium on Corrosion Testing Procedures."

Symposium on Lubricants:

The Wednesday morning and afternoon sessions of the meeting comprised a Symposium on Lubricants sponsored by Technical Committee B on Motor Oils of Committee D-2 on Petroleum Products and Lubricants. The committee immediately in charge of the technical program consisted of the following:

H. C. Mougey (Chairman), Assistant Technical Director and Chief Chemist, Research Laboratories, General Motors Corp.
J. G. Detwiler (Secretary), Assistant Consulting Chemist, The Texas Company.
R. P. Anderson, Secretary, Division of Refining, American Petroleum Institute.
A. L. Clayden, Research Engineer, Sun Oil Co.
L. L. Davis, Supervisor, Process Division, Continental Oil Co.
W. H. Graves, Chief Metallurgist, Packard Motor Car Co.
W. A. Gruse, Senior Fellow, Mellon Institute of Industrial Research.
Raymond Haskell, Industrial Engineer, The Texas Company.
J. L. McCloud, Metallurgical Chemist, Ford Motor Co.
J. B. Rather, In Charge, General Laboratories, Socony-Vacuum Oil Co., Inc.
T. H. Rogers, Assistant Director of Research, Standard Oil Co. (Indiana).
E. W. Upham, Chief Metallurgist, Chrysler Corp.

The purpose of the symposium was to summarize and to bring up-to-date the available information on motor oils and their applications. The present symposium can be considered as a sequel to the 1933 Symposium on Motor Lubricants presented at the fourth regional meeting at New York City, March 8, 1933. It consisted of the following four papers:

"Engine Deposits—Causes and Effects," by W. A. Gruse, Senior Industrial Fellow, and C. J. Livingstone, Industrial Fellow, Mellon Institute of Industrial Research.

"Automotive Bearings—Effect of Design and Composition on Lubrication," by Arthur F. Underwood, Assistant Head, Power Plant Dept., Research Laboratories Division, General Motors Corp.

"Addition Agents for Motor Oils," by George M. Maverick and R. G. Sloane, Standard Oil Development Co.

"How to Select a Motor Oil from the Standpoint of the Consumer," by W. S. James, Chief Engineer, The Studebaker Corp.

The chairmen of the sessions were as follows:

Morning Session.—D. P. Barnard, IV, Assistant Director of Research, Standard Oil Co. (Indiana).

Afternoon Session.—F. L. Faulkner, Manager, Automotive Dept., Armour and Co.

The papers elicited a considerable amount of discussion which contributed greatly to the information contained in the papers. The papers, complete with discussion, have been published by the Society as a separate volume entitled, "Symposium on Lubricants."

The three technical sessions were very well attended, there being about 350 present at each session.

Wednesday Evening Session:

As an innovation from the usual type of informal Regional Meeting Dinner the Chicago District Committee planned an informal dinner at the Casino Parisien which was followed by the regular floor show of the restaurant.

PLASTICS—SOME APPLICATIONS AND METHODS OF TESTING

EDGAR MARBURG LECTURE[1]

BY T. SMITH TAYLOR[2]

DEFINITION

In general a "plastic" is any substance which can be deformed under mechanical stress without losing its coherence. Plastics then include all substances which can be worked and molded into definite shapes under mechanical stress and retain their shape after being formed. This broad definition would include a very large number of classes of materials, such as clays, plasters, all sorts of resins, putty, cement, and even metals. Naturally our discussion will not cover such a broad field but will be restricted to some of those materials which are termed plastics in industry—such as shellac, rubber, synthetic resins, casein products, and cellulose derivatives. All such materials may be considered as being in a plastic state during some part of their manufacturing or their fabricating processes. Certain materials may be considered as plastic under one given set of conditions of pressure and temperature, while under a different set of conditions they may be thought of as lacking this property. In dividing plastics into groups as to origin they may be considered as natural or synthetic.

(*a*) *Natural Plastics.*—Natural plastics include the natural resins, gums, waxes, shellacs, and even rubber, and products made by using them. These substances may be used in their natural state or in combination with other materials. The natural resins are for the most part light in color and vitreous. They are, in general, somewhat hard or at least only slightly sticky at ordinary temperatures. They are capable of becoming soft under the action of heat alone. In general, they are insoluble in water but can be dissolved in certain organic solvents. Natural resins are sometimes called gums or gum resins, particularly by the varnish maker. They are not true gums, however, since gums in general are water-soluble, or at least form gels in water, and are soluble in alcohol.

(*b*) *Synthetic Plastics.*—A synthetic plastic is one made by using a synthetic resin. As the name implies, a synthetic resin is one that is formed by synthesis using as reactants one or more non-resinous organic materials. They may have properties similar to those of natural resins but not necessarily so. They are sometimes spoken of as artificial resins, but the term artificial conveys the idea that the material has been made to imitate the properties of a natural resin. In the case of both natural and synthetic plastics, the term "resin" has been used. The term "resin" was orginally applied to the natural resins arising from plant secretions. A notable exception to this is shellac which is exuded by an insect and not by

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[1] Read on June 30, 1937, before the Annual Meeting of the American Society for Testing Materials, New York City.

[2] Manager, Engineering Laboratory, Diehl Manufacturing Co., Elizabethport, N. J.

a plant. Again the term "resin" has been restricted by some to those resins which can always be rendered plastic by heat. The same authors advocate the use of the term "resinoid" as applied to those resins which may be hardened by heat. For the purpose of this discussion no attempt will be made to use one or the other term exclusively for the purpose of differentiating between the two classes of materials as to the effects of heat upon them. The argument as to the proper terminology to use will be left to the specialists in the resin fields.

KINDS OF PLASTICS AS TO EFFECTS OF HEAT

In considering plastics from the standpoint of the effects of heat upon them, it is convenient to divide them into two groups. The one group embraces those known as thermoplastics, or heat-nonconvertible materials. Such substances are adequately rigid at the temperatures and pressures to which they are normally subjected. They are deformable, however, under the application of heat and pressure. In many cases heat alone will produce a deformation without the addition of pressure. This property of being deformed is practically permanent for this class of materials and can be repeated as often as desired. The other group, according to the effects of heat, contains those materials known as "thermosetting" or "thermohardening," that is, heat-convertible. These "thermosetting" substances originally possess the same properties as the thermoplastic materials. Under the influence of heat, they undergo chemical changes which render them permanently infusible. This property of thermosetting, that is, becoming permanently infusible, varies greatly among the different plastics. In some cases a very short time—say a few seconds—is required to bring about the chemical change, and in others this time is much longer and may be as much as several minutes—and in some cases even days or weeks. Quick thermosetting resins are in demand in most molding operations. In molding thermoplastic materials, it is necessary to cool the mold, to some extent at least, in order that the finished article can be removed from the mold without distortion. Articles molded from thermosetting materials can be removed from the mold while hot with no appreciable distortion. It is obvious that molding operations can be performed more rapidly with the thermosetting types of materials in which the chemical changes take place quickly than is possible with the thermoplastic materials. This statement is based upon the assumption that the time required to bring about the chemical reaction under the action of heat and pressure is small compared with the time required to cool and reheat the mold when using the thermoplastic materials.

It is a matter of fact that the significant difference between thermoplastic and thermosetting materials is becoming of less and less importance. In fact, it has been argued by some that all resins are thermoplastic. It is argued that those which are called thermosetting appear so only because they decompose or dissociate below their softening points. Other technical distinctions and devices of logic so becloud the issue until one realizes that the region is vague in which thermoplastic ends and thermosetting begins. To say that the use of a material depends upon whether it is thermoplastic or thermosetting is too general today. Both types are being modified constantly to meet new requirements. Plasticizers or flexiblizing agents are being added to thermosetting resins so as to prevent the complete formation of infusibility. In many cases radical changes in formulas and manufacturing processes are used to produce the same results.

On the other hand thermoplastic materials are being produced which have higher softening points, decreased temperature susceptibilities, higher plasticities, and other characteristics which cause them to approach the properties of the thermosetting types. In addition to this, there have been marked advances in the use of resins. In order to get specific properties or improve the costs, manufacturers have changed their processes or designs in order to use resins in ways which at one time were considered impossible. Phenol-aldehyde, urea-aldehyde, and alcohol-acid reaction products are extensively used in what are usually spoken of as thermosetting plastics. The raw materials from which these resins are made will, under proper processes of manufacture, produce many varieties of thermoplastics. The cellulose derivatives and the styrene polymers find their main uses as thermoplastics.

SOME PLASTIC MATERIALS AND THEIR PROPERTIES

In presenting some of the chief plastic materials, it should be kept in mind that neither the order in which they are considered nor the emphasis placed on each is to be interpreted as indicating the relative importance or position the material occupies in the plastic field. Furthermore, it is also to be understood that, because no consideration is given to certain plastics, it is no indication that such materials may not occupy even as important places in the plastic field as some of those which are considered. In fact, it may be that there are plastics not herein considered which are decidedly better for their particular field of usefulness than some that are discussed.

Rubber:

It is quite proper that among the plastics some consideration should be given to rubber. In general the name "rubber" and its various forms does not carry with it the idea of its being a plastic as is the case for the materials made using either the ordinary natural resins or the synthetic resins. On the contrary, as a result of the numerous forms and products in which rubber has commercial application, it really has assumed the role of a separate class of material all its own. This is borne out also by the fact that our own Society has for many years had its standing Committee D-11 which has devoted its time to developing methods of test applicable to Rubber Products.

Committee D-9 on Electrical Insulating Materials has been particularly interested in methods for testing the electrical properties of rubber products. As a plastic, rubber may be considered as being used very extensively at all degrees of plasticity from the very soft thermoplastic type to quite hard thermosetting or vulcanized types. That it is possible for the rubber technologist to start with the latex bearing cells of the rubber-bearing tree and alter them so as to produce such a variety of resultant products is really astounding. However, the fact that he must start with the one type of cell does limit his possibilities. It is here where the field of synthetic rubber presents its possibilities. Thus by varying the raw materials and their method of synthesis, a variety of rubber-like substances may be produced, each designed to meet the requirements of a particular field. To a certain extent this is being achieved. The American synthetic product, which is a chloroprene derivative is well known, and complete data concerning its compounding and properties are available. This is not the case for either the

German or Russian synthetic types. Some of these synthetic rubber compounds have excellent physical properties which closely approach those of the natural rubber compounds. In fact, they are much more resistant to oils and gasoline than natural rubber. The synthetic rubbers are somewhat more superior to the natural rubbers in their exposure to elevated temperatures, whereas the reverse is true for low temperatures. By making a shift in the hydrocarbon base, it has been possible to develop a synthetic product which, when properly compounded and cured, neither swelled nor imparted a trace of color to gasoline when immersed in it for a period of months. It is still flexible when submerged in gasoline at a temperature of −35 to −40 F. Recent developments have produced a compound which remains flexible at temperatures as low as −60 F.

From a study of the polysulfide rubbers, a type of synthetic rubber has been produced which can be compounded to any desired degree according to the use to which it is to be put, and then finally cured in any quantity desired in an open steam vulcanizer. This vulcanizate is reduced to a relatively fine powder in a suitable mill. This powder can be molded into the most intricate shapes in a mold in the relatively short interval of 3 min. This oilproof synthetic rubber is now in use in industries in applications where other rubbers are unsuitable. It is quite evident that, as a result of simplicity of manufacturing processes and availability of the necessary raw materials, the polysulfide rubbers can eventually compete in cost with natural rubber products.

Shellac:

Shellac is sometimes called the original thermoplastic. It is quite likely that its use is older than the use of rubber. It possesses unusual hardness, toughness, and durability to wear. Lac resin is the secretion of a small insect living on trees in India and other Eastern countries. The lac resin is collected by natives and purified by crude methods which have undergone no appreciable changes in centuries. In recent years, however, these ancient processes have been carried out by the use of modern machinery and under controlled conditions giving the scientific shellacs which have uniformity and are clean. As much as 25 per cent of the output now is by modern scientific means. Chemically, shellac resin consists of various hydroxy acids which may be partly combined with each other as lactones or anhydrides. There is also present about 5 per cent of natural shellac wax which assists the flow. In addition to this, there will be found about 2 per cent moisture and from ½ to 3 per cent of dirt, sand, and impurities. When shellac is heated above its melting point, volatile matter is given off and condensation or polymerization of the organic acids takes place. This decreases the fluidity of the shellac and raises the softening point so that eventually, if the heating process is continued, it loses its characteristics as a thermoplastic. The rate of polymerization can be increased greatly by the addition of suitable catalysts such as acids, hexamethylenetetramine, urea, benzidine or ammonia salts, and heavy metal oxides or sulfur. By the use of these agents shellac compositions have been prepared which stand temperatures up to 550 F. without appreciable warping and which can be withdrawn hot from the mold. Among the uses for shellac and its compounds may be mentioned: its use as a varnish or protective coating, as a binder for mica flakes, in molded materials, in grinding wheels, and in the production of phonograph records.

Electrically, shellac is an excellent insulator. It does not form a carbon path over the surface after an arc has passed over it. In fact, short circuits have occurred over the surface of shellac insulators and still the insulators remained good for later use.

The general impression is that the consumption of shellac is declining. Relative to the rate of consumption of the newer synthetic insulating materials, this may be the case. Actually, however, the total consumption has increased. In the shipments to America, the amount has increased from a total of about 7,000,000 lb. in 1900 to over 20,000,000 lb. in 1936. Also the total shipments from Calcutta have increased from a total of less than 20,000,000 lb. in 1900 to about 45,000,000 lb. in 1936. Although the increase is smaller than for the synthetic resins, it shows a healthy development in the application of this natural product.

Cold-Molded Plastics:

So-called cold-molded plastics have been in use for many years. They are made using mixtures of asbestos or other fillers with pitch, shellac, cements, and other binders. After being formed to the desired shape by pressure in a mold, they are subsequently baked or heat treated. Cold-molded articles were formerly considerably cheaper than hot-molded ones. The increasing use of hot molding has lowered the cost of production so that the original price advantage does not exist. Some cold-molded materials do possess properties which are superior to the hot-molded ones for certain purposes. Thus there still exist certain applications which will be best satisfied by cold-molded materials, and it is quite likely that such materials will continue to serve a very useful purpose. A recently developed cold-molded material known as "Haveg" promises to fill the special chemical field in which large vessels are desired of special characteristics. As this field is of rather large volume, it will counteract, at least in part, the inroads made in the cold-molded field by the hot-molded plastics.

Cellulose Plastics:

It has been said that the history of the plastics industry dates from the invention of celluloid in 1868 by John Hyatt when seeking a substitute material for ivory billiard balls. He discovered that camphor alone would dissolve pyroxylin, and that thoroughly mixing the two together and pressing the mixture, while under heat, the pyroxylin and camphor would combine into a solid mass. The discovery of the process of making pyroxylin or nitrocellulose is attributed to Schenbain of Switzerland in 1846. Nitrocellulose is formed by treating cotton linters with a mixture of strong nitric and sulfuric acids. After the celluloid is made from the camphor and pyroxylin, it is then seasoned or cured at elevated temperatures in order to remove the volatile alcohol which has performed its function. This curing period may be from a few hours to weeks, or even months. After curing, the surface is finished in any number of different effects. The material may be fabricated in the form of sheets, rods, and tubes of various dimensions. One of the cellulose plastics is cellulose nitrate.

(*a*) *Cellulose Nitrate* or pyroxylin is a true thermoplastic and, by reason of its process of manufacture, innumerable colored and variegated effects are possible. By the medium of dyes and pigments which can be incorporated in the plastic mass, the colorist can obtain many desired effects which are difficult to obtain in other plastics. Since it is readily fashioned by molding, bending, and machining, and possesses highly decorative qualities, it is ideally suitable for such articles as dressing-table sets, fountain pens, pencils, umbrella handles, novelties of all sorts, veneered woodwork, and towel racks. Its toughness, strength, and wear resistance find application in safety glass, heel covers, airplane windows, cutlery handles, brush handles, and many other articles. Among other applications may be mentioned its use in optical parts, piano keys, banjo buttons, organ stops, screwdriver handles, and even zipper fasteners. Pyroxylin plastics can be cemented to themselves by the use of such solvents as acetone and ethyl acetate. They can be cemented to many other articles by use of body cements containing cellulose nitrate, plasticizers, and solvents.

(*b*) *Cellulose Acetate.*—Cellulose acetate constitutes the basic material from which a second type of cellulose plastics known under the same name is made. Cellulose acetate is primarily cellulose, usually in the form of cotton linters esterified with acetic acid and acetic anhydride. It differs greatly from cellulose nitrate in that the nitrate burns quickly and violently while the acetate plastic materials are non-hazardous, burning more like wood, hard rubber, heavy cardboard, and similar materials. The development of cellulose acetate was a real step forward, particularly for picture films, as it rendered the film, which had been previously highly inflammable, perfectly safe. The acetate plastics are marketed in the form of sheets, rods, and tubes for fabricating, and also in the form of blanks and powders for molding. One of the chief applications of the laminated sheet is in safety glass. The stability of the material to light and heat makes it much more satisfactory for this purpose than the nitrate. The acetate alone cannot be readily molded, for it is necessary to add some plasticizing agent. In general, the acetate is more costly than the nitrate, but improved methods of making the acetate plastic in blanks and granular form have greatly reduced the cost till now it is really cheaper than corresponding color sheet of cellulose nitrate. The resistance to impact of cellulose acetate plastics is unexcelled by any other molding composition. The high impact strength and its transparency make it particularly suitable for numerous articles of safety—such as protection goggles, miners' lamp housings, steering wheels, buttons, buckles, and door knobs. The molding compounds, as well as the sheet materials, can be produced in very attractive color effects by the use of suitable pigments and dyes. Translucent and transparent colors can also be produced. The molding compounds can be adapted to both compression and injection moldings. In compression molding it is essential that the mold be chilled somewhat in order that the piece be removed without warping. But even then, cycles as low as 2½ to 3½ min., have been successfully used. In injection molding, even though the material is a true thermoplastic, it is possible to have sufficient heat loss from the mold by natural ventilation and conduction that cooling of the mold is unnecessary. In addition to the items already mentioned which are made from the acetate plastics, a wide variety of articles is produced from the material—such as automobile appointments, bracelets, combs, costume jewelry, knife handles, pencils, switch plates, vanity cases, lamp shades, sash frames, airplane windows, windshields, cockpit enclosures, protective coatings for packages, masks for use in hospital operating rooms, household articles such as pepper and salt shakers, bobbins, and spools.

(*c*) *Ethyl Cellulose Plastics* which constitute another class of cellulose plastics are formulated using ethyl cellulose which has been known in the laboratory for many years but only recently available in this country in commercial quantities. Ethyl cellulose is prepared by the reaction of ethyl chloride on alkali cellulose and is supplied as a white granular powder. Plastics are readily formulated with ethyl cellulose since it is readily soluble in and miscible with many plasticizers and resins. The plastics are normally colorless and transparent. They are unaffected by either alkalies of any concentration or by dilute acids provided the plasticizers used are unaffected. They are little affected by ultraviolet light. They are light in weight, having a specific gravity of 1.14 which is lighter than most molding compounds. Ethyl cellulose is about as flammable as cellulose, but the plastics can be rendered non-flammable by the addition of chlorinated and phosphate plasticizers.

The physical properties of ethyl cellulose give it some distinct advantages in molding plastics. It is readily compounded and produces exceptionally tough plastics. It is lightproof and thus can be used in delicate shades. It is tough even at low temperatures. It can be handled in ordinary molding equipment. It has chemical resistance and good electrical properties.

Phenol-Formaldehyde Plastics:

It may be properly stated that the birth of modern plastics dates from the discovery of Bakelite by Dr. Bakeland some 27 yr. ago while attempting to work out a shellac substitute. The process of manufacturing a typical phenolic resinoid consists of placing weighed quantities of phenol and formaldehyde solutions in a closed jacketed kettle and then heating by means of sending steam into the jacket until the reaction proceeds actively. After the completion of the reaction in a few hours or less, it is found that the contents of the kettle have separated into two parts: an aqueous layer above and a layer of molten resinoid below. This resinoid is soluble in alcohol and when so dissolved, constitutes the varnish with which paper and cloth are treated to form the paper and laminated products. The process of manufacture of the laminated sheets is to press the desired number of sheets, previously treated with the varnish and dried so as to get rid of the solvent, between heated platens in a hydraulic press for sufficient length of time to cause the reaction to be completed—thus thermosetting the resin. When this is completed the resin is no longer soluble or fusible. It is possible to obtain numerous types of this class of resin depending upon the particular raw materials used. Similarly, a large number of types of laminated materials can be secured with any given resin by varying the type of cloth and paper as well as method of manufacture. The manufacture of the molding materials consists of mixing together suitable quantities of resin, filler, coloring matter, and lubricant. The finished articles are fabricated by pressing the desired quantity of molding powder in a hardened steel mold at a temperature of from 275 to 375 F. depending upon the material being molded. The pressures used are also variable depending upon the material and extend over a range of 1000 to 8000 lb. per sq. in. Under the application of pressure and heat, the compound is transformed into the infusible state and can be withdrawn from the mold hot in many cases without fear of distortion. By varying the ingredients, molding compounds of decidedly different qualities can be produced for specific applications. The laminated materials have a wide application in the electrical and mechanical fields. The electrical industry uses immense quantities of laminated materials as insulating materials of construction in transformers, forms for various shaped coils, switch blocks and panels of every description, as well as numerous other applications. Quantities of laminated materials are being used for table tops, wall panelings, and other places in which a decorative effect is desired. The cloth-filled laminated material is used very largely as a gear material and couplings, and particularly so where it is desired to cut down the effects of shock or secure a more silent running machine. In the electrical field use is made of molding compounds for such purposes as wall receptacles, wall plates, connector plugs, twin lights, automotive ignition parts, radio parts, and numerous other applications. In the mechanical group the uses are ever increasing. In recent years emphasis has been placed upon the relative impact strength of the material, and materials of high impact strength have been developed. In general, all phenolic laminated and molded materials are considered as chemically resistant. As an example, where a material must have high tensile strength, high impact value, and also be chemically resistant, may be cited the spinning buckets used in the rayon industry. The fact is that there are so many laminated and molded phenol-formaldehyde parts with which we come into contact daily and we are so accustomed to seeing them that we fail to consider what they really are. For example, in recent years molded compounds have been used to make bottle closures. This application alone consumes hundreds of tons of material yearly. The use of phenolic resins for binders in grinding wheels has produced a wheel of much longer life and more rapid cutting characteristics than was possible previous to this application.

Cast Phenolics:

In the early stages of the development of resinoids, they were used in the cast form. Cast resins differ from the molding and laminated types in that they are poured into molds in their liquid form and then cured in the mold at elevated temperatures. The cast resinoids may vary in properties from a thermoplastic nature to a completely thermosetting character depending upon materials of composition and heat treatment. In general, the molded piece is not the finished product but requires subsequent machining and polishing. The cost of cast phenolic resins is somewhat more than molding compounds. The elimination of expensive mold costs makes it possible for them to compete in many instances with molding compounds. It is possible to so design the molds for certain pieces that the cured piece will require very little if any machining but only require polishing. The largest part of cast phenolic resins is in the form of rods, tubes, plates, and odd shapes for further machining, These materials are used for buttons. brooches, ear-rings, costume jewelry, parts of furniture, and even hardware. Cast resins do not require a filler and can be made transparent or even water white and crystal clear. Clouded and translucent effects can be secured by adding dyes or pigments. Brilliant and delicate shades can be readily produced. The use of such resins has been extending into new fields in recent years. That their use is considerable is evidenced by the fact that in 1936 over 5,000,000 lb. were produced in this country alone. The application of cast resins to dentures has been very successful. The development of these denture materials represents a very extensive series of studies over a period of years in order to develop products which would be especially suited for this application.

Urea-Formaldehyde Plastics:

Although the urea-formaldehyde compounds were introduced many years ago in order to provide lighter colors than were available in the phenol-formaldehyde compounds, they were not offered for sale in this country till about 1929. They can be molded as rapidly as the phenolics but were originally slightly more expensive. The resin itself is clear so that by using highly refined colorless alpha-cellulose pulp as a filler, it is possible to obtain pure white and delicately shaded tints in the finally molded products. The resin is thermosetting and the finished article has a high reflectivity and polish as a result of the depth of color of the compositions. They were not developed to supplant the phenolics but rather to supplement them. Not only can beautiful colors and shades be produced in urea compounds but "light fast" shades are likewise produced. The colors are also "non-bleeding" in alcohol, acetone, and other common solvents. They are odorless and tasteless as is evident from their use in tableware in which hot foods and liquids are used. They are infusible but char at high temperatures. They offer high resistance to arcing over their surface and can be used in electrical equipment where spark-over may occur or where "tracking" causes trouble. They retain excellent insulating properties after immersion in water or contact with damp air. They possess a high degree of flexibility, are high in impact strength and have excellent tensile strength. The ureas are about twice as expensive as the standard brown and black molding compounds but compare favorably in price with the colored phenolics. Among the numerous applications of urea compounds may be mentioned the following: buttons, buckles, closures, jar caps, biscuit and cake cutters, cereal bowls, tableware, bathroom fixtures, bowls for floor lamps, and reflectors. A special application of the urea resins is in the decorative laminated field where they are used for coatings and light colors. In this application their hard surface provides not only decoration but is easily cleaned and withstands ordinary chemicals. Their use in transparent and translucent panels is becoming even more general. In addition to this field, the resin is now finding application in the treatment of textiles and as cements to be used with molded items. The increase in the production of urea-formaldehyde compounds from about 600,000 lb. in 1931 to over 7,000,000 lb. in 1936 shows how rapidly their use is growing.

Furfural-Phenol Resins:

Another resin known as the furfural resin showed definite promise as early as 1920. The high cost of furfural at the time reacted against its commercial use. The resin was developed to meet certain requirements of printing plates. One exceptional property of these resins is their long-drawn-out and flat plasticity curve at the preliminary heating temperatures with little if any polymerization and then a very rapid and almost vertical final polymerization at temperatures above 320 F. This property greatly increases the temperature range over which it can be used. Besides this property of the molding material, the finished piece retains its molded shape with unusual accuracy, as it is of the thermosetting type. The piece can be ejected hot, is strong, and displays little thermoplasticity upon subsequent heatings, indicating that the reaction has been fairly complete. The long-drawn-out stage of plasticity involving little polymerization at the softening temperature permits the use of a dual temperature arrangement in transfer molding. Such conditions permit successive moldings from a given charge in the supply chamber.

Vinyl Resin Plastics:

A resin which was among the earliest of synthetic resins is the vinyl resin. Its history goes back to 1838 when Regnault observed the formation of a white powder when sealed tubes of vinyl chloride were exposed to the sunlight. It was not till 1872 that Bauman polymerized the vinyl halides to white solid masses which were unaffected by solvents or acids. These early discoveries constitute the foundations from which have come the commercial polymerization of various vinyl compounds giving synthetic resins having diversified uses and applications. While these resins are formed by polymerization, they retain their true thermoplastic nature. The vinyl group of resins can be divided into several different series, each series having its own general characteristics and physical properties.

(*a*) One of these series is obtained by the polymerization of vinyl acetate. These resins have relatively low moisture absorption, low heat distortion point, burn slowly, are soluble in most solvents except water, gasoline, and higher alcohols, are colorless, odorless, tasteless, and non-toxic. These resins are widely used as adhesives and have been found extremely useful as an adhesive for cloth, paper, cardboard, porcelain, metal, mica, stone, leather, wood, glass, plastic sheets, and films.

(*b*) A second series of vinyl resins is obtained by the co-polymerization of vinyl acetate and vinyl chloride. These are also colorless, tasteless, odorless, and non-toxic. They possess extreme chemical inertness, being unaffected by alkalies, oxidizing agents, and most acids. These resins are not compatible with nitrocellulose or with most resins or drying oils and, hence, form the sole film-forming constituent when used in the formation of surface coatings. They are affected by long exposure to heat and direct sunlight, but this can be corrected at least in part by the addition of stearates and the commonly used lead pigments. When they are to be used where flexibility is a factor, plasticizers such as the phthalates and tricresyl phosphate are added. These resins are used extensively as lacquer finishes for sheet metal in which case they have to be baked out at relatively high temperatures to insure adhesion. They are also used as impregnating varnishes for cloth, paper, felt, and other fabrics. The impregnated material may be pressed into any desired shape under heat and it will retain this shape. These resins can be fabricated into many applications. Some of these are: dentures of unusual merit, music records for transcribing, sealing containers, closures, poker chips, transparent sun visors, lenses, tooth brush handles, starchless collars, all types of electrical fixtures, and floor tile of unusual beauty.

(*c*) A third group of vinyl resins is an aldehyde reaction product. Probably its chief application is in connection with the manufacture of safety glass, where it produces a glass of exceptional qualities particularly as to breakage at low temperature. The glass is little affected by moisture even after long exposure, does not require sealing at edges after being cut, and is also easily cut. The resin is not discolored by exposure to light, has excellent adhesive qualities, and can be heated to 250 F. so as to permit the baking of enamels on equipment in which it is used.

Acrylic Resin:

The acrylic resins represent another example of a class of resins which have become available through systematic research. The basic acid was prepared as early as 1843, and a polymerized modification was reported in 1877. No serious consideration was given to their commercial possibilities until many years later. Dr. Rohm became interested in their possibilities in 1901 when he published his researches with the acrylic esters. Through Dr. Rohm's interest the acrylic resins were first prepared in quantity in this country in 1931. Their commerical development has progressed rapidly since they were first introduced in 1931. These resins are outstanding in their colorless transparency. They have excellent adhesion; are extremely elastic; and they are chemically resistant to many reagents. Their polymers are relatively unaffected by light, heat, and oxidizing agents, and they range from soft, sticky semiliquids to hard, tough thermoplastics. Various degrees of hardness between these limits are possible by proper modifications during their manufacture without the use of plasticizers. In the advent of acrylic resins, an organic glass came into realization. As a substitute for glass it finds application in lenses, prisms, watch crystals, safety goggles, lighting fixtures, interior decorations, advertising signs, and vehicle windows. Its high impact strength decreases the possibilities of danger from breaking. It is particularly useful in airplane and boat windows. It is readily formed to curved shapes even in two or three dimensions. It does not warp or shrink appreciably with age or with changes in humidity. It is excellent for dentures, fountain pens, spectacle frames, drawing instruments, and novelties. It is not only available in the water-white variety but in many colors. A polymerized derivative of methacrylic acid is also available as a cast resin in the form of sheets, rods, and tubes, and as a thermoplastic molding powder. It is available in both forms as a crystal-clear product and a wide variety of brilliant transparent, translucent and opaque colors. The molding powders require plasticizers and hence have physical properties slightly different from the crystal-clear products. The molding materials can be adapted to either compression or injection molding.

Cumarone Resin:

Brief mention may be made of the cumar resins as thermoplastic materials. They may be obtained from crude coal-tar naphthas in the approximate boiling range of 150 to 200 C. Their development has extended over a period of some 20 yr. They are available in a wide range of colors from faint yellow to darker colors, and in softening points from 10 to 160 C. Being essentially a polymerized hydrocarbon, they may be considered as neutral and stable. The resin is insoluble in water, is not saponified or dissolved in alkalies, has good acid resistance, is soluble in such solvents as benzol and toluol, and has good weathering properties. The resin has properties which make it particularly well suited as a binder in floor tile. It is compatible with the constituents of chewing gum. The grade used has no objectionable odor or taste, and in flexibility and insolubility it meets all chewing gum requirements. It is an important constituent in rubber compounding since it blends well with rubber and facilitates the milling operations. Its electrical properties make it well suited for electrical insulation service. It is also very useful in varnishes and particularly suitable for aluminum paint formulation. It has proven useful in printing ink varnishes. Road marking paints using this resin stand up very well. It is also used in the adhesive industry. The resin blends well with waxes yielding many solids of considerable possibilities. It can also be combined with other synthetic resins and thus produce products which meet definite requirements not met by the resin with which it is combined. It is quite likely that the use of cumar resin will amplify and supplement the thermosetting resins in both molded and laminated products.

Polystyrene:

Another resin which is among the oldest of the synthetic resins is Polystyrene. Its development has lagged behind resins of poorer qualities as a result of the lack of suitable manufacturing facilities and methods. These drawbacks have been recently removed through systematic studies. Polystyrene is derived from styrene a colorless liquid by the process of polymerization with or without calalysts. Styrene can be produced in a variety of ways. Probably the most satisfactory way is the process by which two hydrogen atoms are removed from ethyl benzene by pyrogenic dehydrogenation or by chlorination and later removing the hydrochloric acid. It has a defect, so to speak, of polymerizing at room temperature when exposed to sunlight unless prevented from doing so by inhibitors. Its physical properties are largely determined by the conditions under which it is polymerized. It may be made so as to be extremely tough or quite brittle as desired. Polystyrene can be molded under the application of heat and pressure and, being a true thermoplastic, continued application of heat at molding temperatures does not affect it. It is chemically inert and has remarkable electrical insulating properties, its dielectric losses being superior to those of porcelain and vastly superior to other resinous insulating materials. It also has good resistivity and dielectric strength. Its softening point is about 10 deg. Cent. higher than that of the usual hard rubber. It will retain its shape in the molded piece up to 110 C. unless external strain is applied. Polystyrene, being itself colorless and transparent, can be easily colored to any desired tint. It can be used with the ordinary fillers up to 40 per cent by volume. When necessary it can be plasticized by use of dibutyl phthalate and many of the usual plasticizers. It is not compatible with rubber, shellac, cellulose esters, or ethers, and it does not give clear moldings with these materials. It is compatible though with cumarone resins. Polystyrene is well suited for dentures and, on account of its excellent electrical properties, for radio parts and similar applications. With continued improvements in methods and lower costs of materials its production is due to increase.

Casein Plastics:

Casein plastics were produced commercially in Europe as early as 1904. They are made from the casein obtained from milk, and after it is pressed into sheets or rods it is cured or seasoned in a formaldehyde solution. The sheets or rods are then dried and straightened. This curing and subsequent drying takes considerable time, requiring six weeks for sheets $\frac{1}{4}$ in. thick and about six months for rods over $\frac{5}{8}$ in. in diameter. The usual sheet is 16 by 20 in. and of thicknesses under $\frac{1}{2}$ in. Tubing is also made but if the bore of the tube exceeds one-half the outer diameter, the tube is likely to become oval while it is being extruded. Casein products are somewhat hygroscopic, and pieces larger than 4 in. square are likely to warp. The most important use of casein is for small articles where color, appearance, and durability are the first considerations. When molded to shapes of varying dimensions and thicknesses, it cures more readily in the thinner sections than in the thick and hence gets out of shape when removed from the mold. It is for this reason that casein has never been made available as a molding powder. Casein plastics are made in a wide range of opaque and translucent plain colors, and many mottled effects. They are made so as to imitate horn, metallics, mosaics, jades, precious stone effects, and even pearl. The material can be machined readily and takes a fine and lasting polish. Its principal use in this country is for buckles, buttons, slides, and accessory trimmings, and to some extent in the manufacture of electrical equipment and appliances.

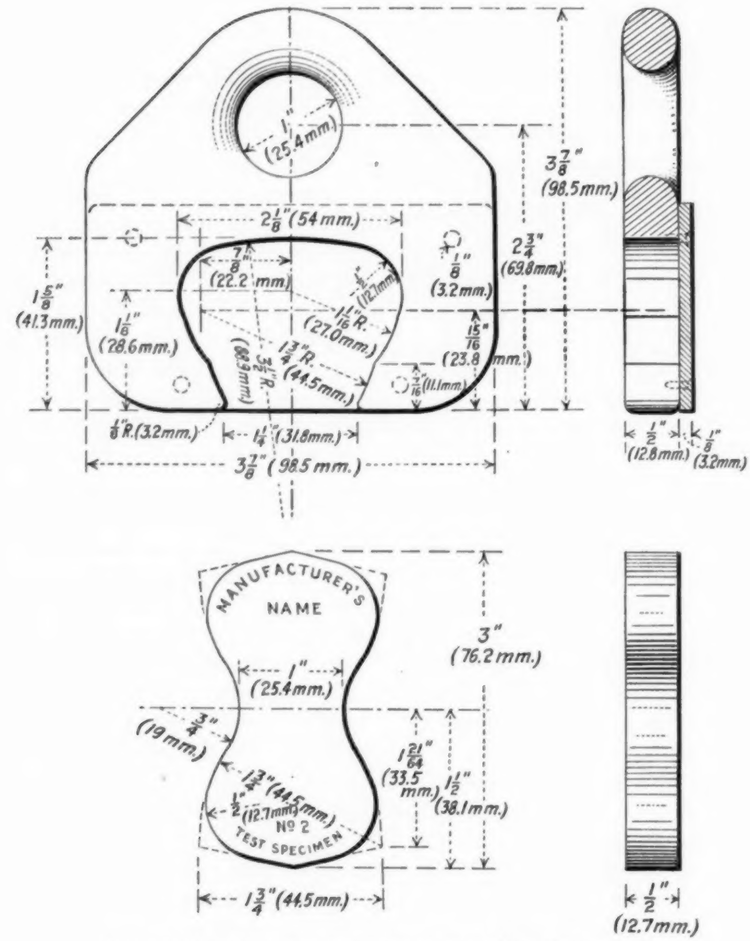
OTHER APPLICATIONS OF PLASTIC RESINS

Besides the use of resins in molding compounds, laminated materials and applications already discussed, there are many other places in which their commercial importance is ever on the increase. Among these mention may be made of the following: some of the resins are used as a basis of the heat hardenable varnishes, enamels, lacquers, cements for binding different materials together, lamp basing cements, and bristle setting cements. Other resins constitute the basis of many air-drying varnishes, lacquers, and finishes. Again the use of synthetic resins in connection with improving the properties of paints is constantly increasing. The application of synthetic resins to these fields has increased from only small quantities a few years ago till at the present time the amount of resin used in all the paints, varnishes, and finishes constitutes a reasonable fraction of the resins produced.

It has only been possible to speak of a few of the many types of plastic resins that are now available commercially. During the past 20 yr. the number of useful ones has increased through the efforts of systematic research from a few till now there is a host of them each having certain specific properties differentiating it to a more or less extent from all others. At first the application of synthetic resins to plastics and other products was somewhat of a specialty or novelty. This has now taken on an entirely different aspect in that they now constitute an integral part of our industries and promise to become of ever increasing importance in the future.

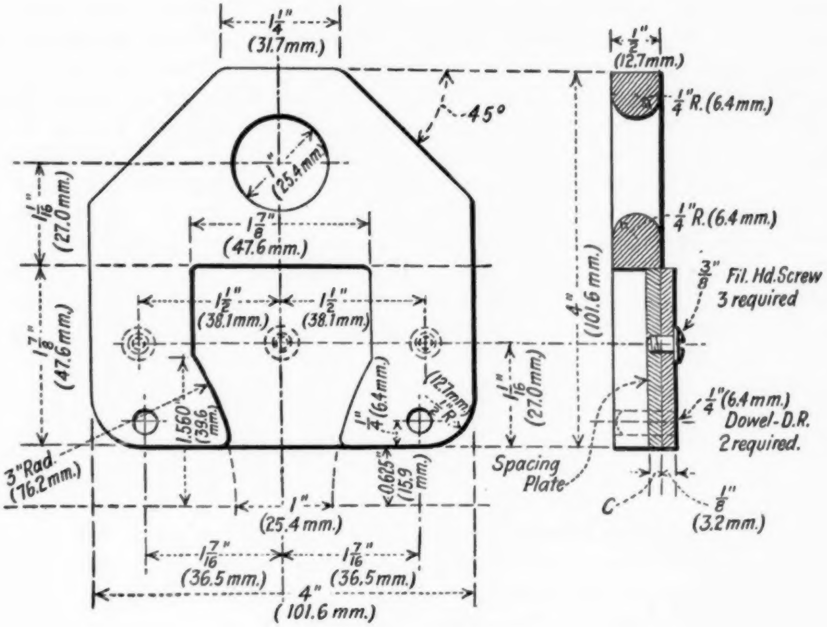
TESTING OF PLASTICS

Originally it was planned that a considerable portion of this discussion would be given to the consideration of methods of testing plastics. In order that it not be unnecessarily prolonged, however, I will limit what I have to say on methods of testing to a brief consideration of the development of the method of testing the tensile strength of molding compounds, as furnishing an example of the process by means of which our methods of test are shaped into their existing forms. Due to the introduction of the phenolic compounds into the electrical industry, interest arose in the developing of methods of testing their properties, among which was tensile strength. The question arose as to the form of specimen to use to determine tensile strength and also how this specimen should be held in the tension testing machine. Those interested in testing cements had already designed a figure-eight shaped specimen which could be formed in a suitable mold, and in being tested it could be held in specially constructed holders instead of being clamped in friction jaws. As this same shaped piece could be readily made from molding compounds in a suitable mold, it was used as a tension test specimen for these materials for several years during the early growth of the phenolic molding industry. The specimen and holders are shown in Fig. 1. The specimen had a uniform thickness of $\frac{1}{2}$ in. and was 1 in. wide at the narrowest part changing immediately on both edges by sharp curves to the wider ends. In testing, the specimen rested in suitable holders on the curved portions of the edges so that the piece and holders were practically self aligning. In general, the piece broke at its smallest cross-section. An occasional break for certain materials occurred in the wider portion under the point on contact of the holders. Indications that the values of tensile strength obtained by this specimen were low came from two different sources. The first of these was the fact that the tensile strength of molded materials which had been obtained by a type of specimen in the form of a spool having narrow circular cross-section at its middle point were always considerably higher than those obtained by means of the flat figure-eight specimen. The other one arose from work that had been undertaken to determine the tensile strength of laminated materials. It had been the custom to determine the tensile strength of laminated materials using a



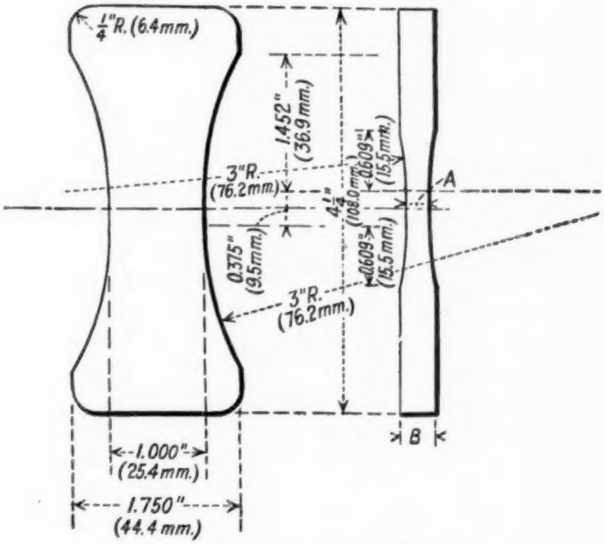
Make Steel Mold to these Dimensions. Limits ± 0."002 (0.05mm.)

FIG. 1.—Tension Test Specimen.



$1\frac{1}{4}$" (31.7mm.)
45°
1" (25.4mm.)
$1\frac{1}{16}$" (27.0mm.)
$1\frac{7}{8}$" (47.6mm.)
$1\frac{7}{8}$" (47.6mm.)
$1\frac{1}{2}$" (38.1mm.)
$1\frac{1}{2}$" (38.1mm.)
1.560 (39.6 mm.)
$\frac{1}{4}$" (6.4mm.)
(12.7mm.) R.
$1\frac{1}{16}$" (27.0mm.)
3" Rad. (76.2mm.)
0.625" (15.9 mm.)
1" (25.4mm.)
$1\frac{7}{16}$" (36.5mm.)
$1\frac{7}{16}$" (36.5mm.)
4" (101.6 mm.)
4" (101.6 mm.)
$\frac{1}{2}$" (12.7mm.)
$\frac{1}{4}$" R. (6.4mm.)
$\frac{1}{4}$" R. (6.4mm.)
$\frac{3}{8}$" Fil. Hd. Screw 3 required
$\frac{1}{4}$" (6.4mm.) Dowel-D.R. 2 required.
Spacing Plate
C
$\frac{1}{8}$" (3.2mm.)

(*a*) Specimen holder.



(*b*) Tension test specimen.

FIG. 2.—Tension Test Specimen and Holder.

test specimen of uniform width cut from a sheet of the material. For the straight pieces, it had been observed that the specimens broke more frequently either within the grips or close to them rather than nearer the middle portion of the piece. In order to see whether the grips were actually affecting the values for the laminated materials, test specimens were prepared which had a portion having a straight section of uniform width say 1 in. at its middle and then changing to wider end sections by various radii of curvature from $\frac{3}{4}$ to 3 in. With this type of specimen, consistently higher values were obtained for the tensile strength of laminated materials than had been obtained using the straight piece of uniform width throughout. In addition the specimens always broke either within the narrower section at the middle or where the curvature to the wider section began. The values obtained with the pieces having the 3-in. radius of curvature were consistently the highest and most uniform. These facts being observed for laminated materials, the question arose as to whether the jaws holding the figure-eight molded piece were not influencing the results. To test this, specimens were prepared by machining which had a long section at the center of uniform thickness and width and then changed to a wider section at both ends so as to be held in the regular jaws for the figure-eight specimens. These specimens always broke near the holders and gave values no higher than the figure-eight specimens. Then specimens were prepared having larger radii of curvature so that the change from the uniform narrow middle portion to the wider ends was more gradual. These specimens gave consistently higher values than had been obtained previously. These observations definitely indicated the fact that the tensile strength of the figure-eight specimens was being affected by the compressive action of the holders on the edges of the specimen. All these results indicate that in order to obtain tensile strengths of molded materials which would be unaffected by the methods of holding the specimen during the test, it would be necessary that the specimen have a reduced section where the break was to occur at its middle portion which would change by as large a radius of curvature as possible to a much heavier section at each end where it was to be held. Then the specimen must be such as to be readily molded.

Further studies led to the fact that if the piece was to be kept relatively short, it would have to be made with a change in thickness from the reduced section at the middle as well as a change in width in order to insure a break which would not be influenced by the compressive action of the holders. The present tension test specimen is the result of these studies. The specimen and holders are shown in Fig. 2. This specimen has a uniform sectoin $\frac{3}{8}$ in. long, 1 in. wide, and $\frac{1}{4}$ in. thick at *A* its mid-portion The thickness then changes by means of 3-in. radii of curvature on each side from this $\frac{1}{4}$-in. thickness at the middle to a uniform thickness of $\frac{3}{8}$ in. at each end, *B*. The width also changes from 1 in. at the middle by means of 3-in. radii of curvature on each edge till the width is $1\frac{3}{4}$ in. at each end. The specimen is held in special grips which allow the specimen to rest its wider portion on its edges in holders made to fit the 3-in. curvature. Held in this way the specimen is practically self aligning and the compressive action of the grips has no influence on the strength obtained as they are well removed from the section of smallest area where the breaks occur. The curvatures of 3 in. from the central reduced section to the wider ends do not completely remove the possibility of there being a non-uniform distribution of stress in the reduced section at the time of break. The ideal specimen for such tests would be one that is circular in cross-section at the reduced portion at the center, and would change by large radii of curvature to ends of considerably larger area of cross-section and which could be held in satisfactory self-aligning jaws at the ends. However, such a specimen is not well suited for all types of material though it would be quite satisfactory for materials that could be molded by injection molding. The present specimen is then a sort of compromise so as to be molded relatively quickly. While this tension test specimen has proven to be quite satisfactory for the materials for which it was originally developed, it is not at all impossible that its continued use even in testing such materials and especially its application to the newer plastics may lead to information which will result in still further modifying the specimen or in fact to the development of entirely different ones adaptable particularly to given materials.

In a similar manner, the development of the test specimens used in the determination of other physical properties of plastics could be outlined. In the development of the tension test specimen, it was unnecessary to give much consideration to the method of test since there was an unlimited amount of experience available in connection with methods of determining tensile strength. This has by no means been the case with other methods of test such as impact, dielectric strength, resistivity, and power factor. In some of these the development of the method such as that for power factor has been far more important and difficult than has the determination of a suitable specimen.

SIGNIFICANCE OF TESTS

Before closing I should like to make a few statements relative to the significance of tests. We are all interested in being able to obtain as nearly an accurate value of the physical and chemical properties as possible. We wish to know what is the proper value to be ascribed to a given property of a material. Having once established this, then the question arises as to just what do the results signify to us as consumers and as producers. In some respects they will mean the same to both consumer and producer. In others the test results may be used quite differently. Thus the producer can use the results of his tests to guide him in maintaining uniformity in his product. He can use the results for determining the uniformity and constancy in his raw materials, for determining whether his processes are sufficiently controlled, for determining the relative merits of different raw materials, for determining the suitability of his product for specific uses, and for the improvement of both processes and products. The consumer on the other hand needs to know the various properties of the articles he is purchasing in order that he may be sure they will meet the requirements placed upon them in service: that he may know whether the article will function properly in a given application, that he may be guided in substituting a so-called cheaper or better article for one now in use which has proven satisfactory. An intelligent interpretation of test results on the part of both consumer and producer, will result in improved products, less expensive products, and more satisfactory functioning of equipment and apparatus in which they are used.

While this discussion has only touched upon a few of the phases of plastics and their applications, it is evident that plastics and plastic compounds occupy a very important place in our every-day life and promise through continued research to be of ever-increasing value to our civilization.

WELD METAL AS AN ENGINEERING MATERIAL AND SOME METHODS OF TESTING

By L. J. Larson[1]

Synopsis

This paper discusses the physical tests, chemical analyses and metallurgical studies used in the investigation of weld metal. In addition to standard tests it has been necessary to develop new types of tests and to modify standard test procedures to obtain the desired data on weld metal. As a result of chemical and metallurgical studies, the factors governing the quality of weld metal are known. With this knowledge it is possible to control the welding process so as to produce weld metal that is uniform and reliable as an engineering material.

In the various fusion-welding processes the edges of the parts being joined and generally some additional filler material are fused together to form the joint. This fused metal is called weld metal. In this paper weld metal will refer to the fused metal produced by the metallic arc with either bare or covered electrodes.

Although the metallic arc process is old, weld metal as an important engineering material is relatively new. With the bare electrodes formerly used the quality of the weld metal was suitable only for making repairs or for joining light material. The stresses to which the welds were subjected in these applications were often very small and an accurate knowledge of the properties of the weld metal was unimportant. With the perfection of covered electrodes within the past fifteen to twenty years the metallic arc process has been developed from a repair tool to one of the most important methods of joining metals, both in the factory and in the field.

In the various applications of welding, the weld metal is subjected to the same kinds of stresses as the material being welded, that is tension, compression, and shear. The loads may be static or they may be alternated or repeated, and they may be applied slowly or with impact. The structures may operate at room temperature, at temperatures as high as 1000 F., or at subzero temperatures. In a structure such as a pressure vessel in which the shell sections are welded with butt joints of the same thickness as the shell, the weld metal is called upon to withstand the same stresses and operating conditions as any portion of the plates from which the vessel is constructed. Inasmuch as the weld metal is subjected not only to the same kind of stresses as the base metal but to stresses of as great or greater magnitude, it is just as important to know the mechanical properties of the weld metal as to know the mechani-

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cal properties of the material being welded.

METHODS OF TESTING GENERALLY USED

In the study of weld metal, physical tests, chemical analyses, and metallurgical examinations have been employed. It has been possible to use many of the standard tests, but it has also been necessary to develop some new tests or to modify standard test procedures to obtain the desired data on weld metal. Some of the physical tests used are designed to determine the properties of the weld metal and others to investigate the strength of the welded joint.

Physical Tests:

The determination of the properties of weld metal requires specimens consisting entirely of weld metal and offers some difficulties not encountered in the study of other materials. Most metals occur in the form of castings, plates or bars from which test specimens can be readily obtained. Weld metal is deposited in beads which may be triangular in cross-section as in fillet welds or either triangular or nearly rectangular in cross-section as in butt joints. When the cross-section is large enough, a standard ½-in. round specimen of weld metal can be turned and the yield point, tensile strength, elongation, and reduction of area of the weld metal can be determined by the usual methods. If the weld is too small to obtain a ½-in. round specimen, a flat tension specimen, taken parallel to the weld and consisting largely of deposited metal with a small amount of plate material along the sides or edges, may be used. Tension tests on such specimens give good approximations of the values for the properties of the weld metal. A rectangular tension specimen parallel to the weld and wide enough to include all of the stock affected by the welding and some unaffected plate material is useful in indicating the relative ductility of the weld metal and the adjacent plate material. When such a specimen is tested, if the weld metal is brittle, it will crack and start a tear in the plate material which may result in an ultimate load considerably below the combined strength of the weld and the plate material. Under certain conditions some types of material may be hardened or embrittled adjacent to the weld and the fracture may start in the affected zone of the plate. This type of test indicates the presence of a brittle zone even when the material in such a zone has ample strength, whereas a test of a tension specimen across the weld may not reveal this condition. Failures in service of brittle weld metal joining mild steel plates which were subjected to tensile stresses parallel to the weld have occurred.

The strength of a welded joint is generally determined from flat tension specimens taken across the weld. When such specimens have a parallel section extending several inches on either side of the weld, the failure may occur in the stock instead of in the welded zone. The values obtained from such specimens are not, as a rule, the properties of the weld metal. The yield point obtained by the drop of the beam is likely to be the yield point of the stock since the weld metal usually has a higher yield point than the plate material. By the divider method the yield point of the weld metal can also be obtained if the gage points are located in the weld. The tensile strength of the weld metal is obtained only when the fracture occurs in the weld. The percentage elongation is a composite value for stock and weld metal unless the gage line used is short enough to include only one or the other. When failure occurs in the stock, the elongation value obtained from such specimens is no indication of the ductility of the weld metal. A modified tension specimen, known as a reduced-section tension specimen,[2,3] has been developed to obtain more information on properties of weld metal. In this specimen, which is taken across the weld, the length of the parallel section is only slightly greater than the width of the weld, and the failure must occur in the weld or very close to it. The tensile strength of the weld metal may thus be obtained even when it is somewhat stronger than the stock. This test is also very effective in disclosing any weakness in the fusion zone if such weakness exists.

In order to obtain more information as to the ductility of weld metal a test known as a free bend has been developed.[2,3] The specimen for this test is taken across the welded joint with the weld in the middle and has a width of $1\frac{1}{2}$ times the thickness of the plate. The weld reinforcements are removed and the corners of the specimen are rounded to a radius of about $\frac{1}{16}$ the thickness of plate. The specimen is kinked or bent near the third points after which it is set up in a testing machine as a column. As the load is applied the specimen gradually bends into the form of a U. The elongation of the weld metal is obtained from measurements of the change in length between gage points located in the weld on the outer surface of the bend. Most of the welding is generally from one side of the plate, and this side is made the outside or tension side of the bend specimen. In many cases a second specimen of the same dimensions, called the reverse bend, is bent in the opposite direction to show the ductility of the bottom of the weld and to indicate whether fusion at the bottom of the weld is complete.

Other physical tests to determine the properties of weld metal include fatigue tests, impact tests, and tension tests at elevated temperatures. These are generally not required as acceptance tests in connection with a particular structure but are part of a general study of weld metal.

In addition to the physical tests mentioned above which are of a quantitative nature, a number of qualitative tests have been devised for checking the soundness of welds. Lack of soundness may be due to porosity or gas pockets, to slag inclusions, and to cracks. The tests for detecting these defects may be divided into two general classes: destructive and non-destructive tests, the latter being applied to welds in the finished structure.

Two destructive tests have been used to a considerable extent: the nick break test and the specific gravity test. In the nick break test a specimen having about the same dimensions as the bend test specimen is supported as a beam and broken through the weld by a sharp blow. In order to cause the fracture to take place in the weld a saw cut or V groove is made in the weld at each edge of the specimen and the specimen is placed on edge on supports so these notches are on the top and bottom of the test specimen, respectively. From a visual examination of the fracture conclusions can be drawn as to the degree of porosity and other defects. The presence of coarse crystalline and brittle zones in the weld is also shown by this test.

In the specific gravity test the density of the weld metal is determined either directly by immersion in water or by computation from the weight and dimensions of the specimen. Some weld metal, such as that made with bare wire,

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[2] See A.S.M.E. Code for Unfired Pressure Vessels, p. 61 (1935).

[3] A.P.I.-A.S.M.E. Code for Unfired Pressure Vessels, p. 61–63 (1936).

which has a fine dispersion of impurities and small porosity may appear sound on a machined surface and still have a specific gravity as much as 3 per cent below that of steel. As a part of a program of studying weld metal, the specific gravity test serves to distinguish between types of welds which have a uniform and general distribution of inclusions or voids and types which are free from such defects. As an inspection test it is less searching than the usual physical tests because it explores only a very limited amount of weld metal and the specimen is located in the portion of the cross-section which is least likely to contain the more serious defects such as slag inclusions and lack of fusion. With the modern types of weld metal, the uniformly distributed porosity is not much of a problem. When such porosity does occur, the gas pockets are large enough to be easily visible in the specimens for the physical tests and the specific gravity test adds very little information not shown by other tests.

Three non-destructive tests have been used. In one of these the defects in the welded joint are indicated by disturbances in the magnetic field as shown by the arrangement assumed by iron filings or iron powder when the joint is magnetized. This method, especially when iron powder is used, is sensitive in locating cracks which come to the surface. For defects below the surface it indicates only relatively large voids. A second method of locating defects is to listen with a stethoscope to the sound vibrations set up by tapping the structure with a hammer. Relatively large defects such as cracks and lack of fusion are detected by this method but it does not indicate porosity. The third method is radiographic examination by means of radium or X-ray equipment.[4] Modern X-ray machines are capable of penetrating steel up to 4 and possibly 5 in. thick with a sensitivity sufficient for practical purposes. Radium can be used on much greater thicknesses, but the sensitivity is less on these heavier sections. Radiographs reveal the presence of all types of defects such as porosity, slag inclusions, lack of fusion, and cracks. With physical tests to indicate the properties of a given type of weld metal and radiographs to insure that the welds in the structure are sound, uncertainties as to the properties of the welds in a structure are eliminated. However, radiographing is relatively expensive and is, therefore, used only on welds in structures subjected to hazardous service conditions.

Chemical Analyses:

The elements which determine the properties and behavior of mild steel are: carbon, manganese, phorphorus, and sulfur. The methods of analyzing commercial steels for these elements are well known and the same methods are used in analyzing weld metal. In addition to the above-mentioned elements, weld metals may contain silicon, nitrogen, and oxygen in significant amounts. The methods of analyzing for silicon and nitrogen are well established and can be carried out in any well-equipped laboratory. For the determination of oxygen the vacuum fusion method is the most satisfactory process, but it requires special equipment which is not available in many laboratories. Although the process was developed to analyze commercial steels, its use in the study of weld metal has resulted in modifications and improvements in the process.

The equipment used for the vacuum fusion process consists essentially of a small induction furnace connected to suitable pumps for evacuating the system and reservoirs for collecting the

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[4] See Symposium on Radiography and X-ray Diffraction Methods, Am. Soc. Testing Mats. (1936). (Available as separate publication.)

gases given off by the test sample. The specimen to be analyzed, a cylinder about 11 mm. in diameter and 35 mm. long, is carefully cleaned, weighed, and placed in a holder in the head of the furnace. With the system, including the furnace, sealed gas tight, the furnace is heated to the desired temperature for melting the sample and the system is evacuated to 0.05 mm. mercury or less. The specimen is then dropped into the furnace and the gases given off by the molten steel are collected. This gas sample is analyzed to determine the percentages of oxygen, nitrogen, and hydrogen.

TABLE I.—MECHANICAL PROPERTIES OF WELDS AND PLATE.

Type of Electrode	Plate Thickness, in.	Yield Point, lb. per sq. in.	Tensile Strength, lb. per sq. in.	Elongation in 2 in., per cent	Reduction of Area, per cent	Bend Elongation, per cent	Charpy Impact, ft-lb.
Bare...........	$\frac{1}{4}$ (as welded)	40 000 to 45 000	50 000 to 60 000	6 to 15	....	15 to 30	....
Bare...........	1 (stress relieved)	20 000 to 45 000	25 000 to 55 000	1 to 7	0 to 7	1 to 12	1 to 2
Covered........	$\frac{1}{4}$ (as welded)	40 000 to 55 000	55 000 to 70 000	20 to 25	....	30 min.	....
Covered........	1 (stress relieved)	40 000 to 55 000	55 000 to 70 000	25 to 40	40 to 70	30 min.	25 to 45
Covered........	Over 1 (stress relieved)	40 000 to 55 000	55 000 to 70 000	25 to 40	40 to 70	30 min.	25 to 45
0.20 to 0.30 per cent carbon steel plate		30 000 to 40 000	55 000 to 65 000	35 to 50	50 to 70	....	20 to 30

A modification of the vacuum-fusion process, known as the "fractional vacuum fusion," has been developed by Reeve.[5] Taking advantage of the fact that the oxides, FeO, MnO, SiO_2 and Al_2O_3 are reduced by carbon at different temperatures, Reeve worked out a procedure for separating the oxygen into three fractions, namely, FeO, MnO; SiO_2; and Al_2O_3. Although he did not attempt to give the percentages of FeO and MnO separately, he observed that FeO comes off more rapidly and at a lower temperature than MnO and it is thus possible to estimate roughly the amount of each. The information obtained from the fractional method has done much to explain the behavior of weld metal.

Metallurgical Examination:

The methods used in the metallurgical study of weld metal have been the same, in general, as those used in the study of commercial steels. Some special heat treatments and tests have been used to bring out certain characteristics of the metal. Among these are the "nitride anneal" to precipitate the nitrogen when present in sufficient amounts; hot shortness tests to indicate excessive FeO; and the Fry test to indicate an aging metal.

RESULTS OF TESTS

Physical Tests:

Mechanical properties of welds made with bare and with heavily covered electrodes are given in Table I. For purposes of comparison, values are also given for 0.20 to 0.30 per cent carbon plate material. For welds in $\frac{1}{4}$-in. plates the results are given for the as-welded condition, that is without stress relieving since structures built of material of this thickness are seldom stress relieved. For the welds in the heavier plates, the values given are for the stress-relieved condition.

The yield point, tensile strength, elongation and reduction of area of welds in plates 1 in. thick and heavier were obtained on $\frac{1}{2}$-in. round specimens of weld metal. The bend elongations were

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[5] Lewis Reeve, "Improvements in Vacuum Fusion Method for Determination of Gases in Metals," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 113, p. 82 (1934).

obtained by the method previously described, on flat bars taken across the weld. The keyhole notch type of specimen was used for determining the Charpy values. For welds in ¼-in. plates the values given were obtained on specimens taken along the axis of the weld. The bend specimens and the reduced section of the tension specimens were ¼ by ½ in. in cross-section.

The results for welds in plates 1 in. and heavier made with covered electrodes are based on tests of well over a hundred specimens of each type except the impact values which are based on about twenty tests. Many of the specimens used in these tests were from production welds in material of various thicknesses up to 4½ in.

The number of specimens tested of welds made in 1-in. plates with bare wire was not so great. Bare wire is not used in production for welding plates as heavy as 1 in. and the tests were limited to welds made specifically for test purposes. Five tension specimens of weld metal and eight specimens across the weld were tested. Since failure of the specimens across the weld occurred in the weld with no apparent deformation in the plate material, these specimens furnished data on the ductility and reduction of area as well as the strength of welds made with bare wire. Eight bend specimens and eight Charpy bars were also tested.

From the welds made in ¼-in. plates with bare and with covered electrodes, two tension and two bend specimens taken along the axis of the weld were tested. A number of other specimens taken across the weld showed that the welds made with bare and with covered rods both had tensile strengths equal to the strength of the plate used, or about 56,000 lb. per sq. in.

A study of the values given in Table I shows a number of interesting and significant facts concerning weld metal. Welds made with bare wire in ¼-in. plates have a yield point and tensile strength comparable with those of mild steel but the ductility values are rather low. In 1-in. plates the yield point and tensile strength may be fairly high in some specimens, but in other similar specimens the strength may be very low and there is no true yield point. The elongations in both the tension and the bend specimens, the reduction of area, and the Charpy impact values are all low and in some cases may be nearly zero.

Welds made with covered electrodes in all three thicknesses of plate have a yield point higher than and a tensile strength equal to or higher than the corresponding properties of mild steel. In ¼-in. plates the ductility of the welds without stress relieving is lower than that of the plate. Welds in heavier plates have elongation and reduction of area values comparable to those of the stock, and the Charpy impact values are higher than those of the plate material. The values given for the bend elongations of the weld made with covered electrodes are not the elongations at rupture. In the routine bend test of production welds the test is stopped when the elongation reaches 30 per cent, since this is all that is required by the various codes. On specimens carried to failure, elongations of as much as 100 per cent have been obtained. In some cases, specimens may bend flat on themselves without failure. It will be noted that neither the strength nor the ductility of welds made with covered electrodes decreases as the thickness of the stock increases. Welds in plates as thick as 6 in. have shown strength values greater than those of the stock. This is significant from a practical standpoint and indicates that this type of welding is suitable for the range of thicknesses of plates commercially available.

Other types of physical tests have shown that weld metal made with properly covered electrodes has properties similar to mild steel. The long-time tensile strength at 900 F. of such weld metal is about 18,000 lb. per sq. in., which is also the strength of 0.20 to 0.30 per cent carbon steel. Fatigue tests show that the endurance limit of welds made with covered electrodes and of mild steel are 30,000 lb. per sq. in., which is about twice the endurance limit of welds made with bare wire.

TABLE II.—CHEMICAL AND GAS ANALYSIS AND MECHANICAL PROPERTIES OF WELDS AND OF PLATE MATERIAL.

	Weld Made with Bare Wire	Weld Made with Covered Electrode	Plate Material
Carbon, per cent	0.02	0.08	0.19
Manganese, per cent	0.09	0.57	0.56
Silicon, per cent	0.01	0.25	0.01
Phosphorus, per cent	0.012	0.012	0.028
Sulfur, per cent	0.027	0.023	0.022
Oxygen, per cent	0.298	0.072	0.024
Nitrogen, per cent	0.131	0.010	0.004
Yield point, lb. per sq. in	44 200	44 300	35 000
Tensile strength, lb. per sq. in	53 800	57 500	56 500
Elongation in 2 in., per cent	6.0	37.5	45.0
Reduction of Area, per cent	6.3	68.7	63.9

In addition to the bare and covered electrodes previously discussed, there have been developed and used a number of types of electrodes with dusted, washed, and lightly dipped coatings. The mechanical properties obtained by the use of such electrodes range from those of bare wire welds at one extreme to those of welds made with covered electrodes at the other. The properties of the weld metal produced by these various electrodes depends upon the effectiveness of the coating in protecting the metal while it is being deposited as will become more evident from the results of chemical analyses and metallurgical studies.

Chemical Analyses:

The behavior of various types of weld metal is largely explained by the chemical analyses of the deposited metal. Analyses and mechanical properties of one weld made with bare and one with covered electrodes and of the mild steel in which the welds were made are shown in Table II. Referring to Table II it will be seen that the weld made with bare wire is low in carbon and manganese. Without further information this metal would be expected to be soft and ductile and quite similar to pure iron. Silicon, phosphorus and sulfur are present in about the same amounts in this weld metal and in mild steel and should have no more effect on the weld than on the plate. A comparison of the oxygen and nitrogen contents of weld metal made with bare wire with those of mild steel shows a striking difference. This weld metal has about twelve times as much oxygen and thirty-three times as much nitrogen as the steel plate.

Comparing the analyses of welds made with covered electrodes with those of the plate, it is noted that the carbon in the weld is less than one-half as much, and the manganese, phosphrous, and sulfur are about the same as in the plate. The silicon in the weld corresponds to that in a silicon killed steel. This amount of silicon has very little effect on the properties of steel except for its killing action. The oxygen content of this weld is three times and the nitrogen content two and one-half times as much as that of the plate material.

It is generally understood that both oxygen and nitrogen may exert pronounced effects on the properties of steel when present in sufficient amounts. An excessive amount of oxygen makes the steel hot short within a certain temperature range and a high nitrogen content causes the material to age harden.

Both oxygen and nitrogen tend to increase the strength and decrease the ductility of steel at room temperature.

Hoyt[6] found that a heat of pure iron which was oxidized during melting was hot short. He also found that weld metal made with bare wire was hot short and that made with covered rods were not. In the strain aging test he found that the former was etched dark by the Fry reagent and the weld made with the covered rod showed no Fry lines, indicating that the former is aging and the latter is non-aging. Hensel and Larsen[7] concluded from their tests that the increase in hardness of arc welds during aging is dependent upon the nitrogen content and that the nitrogen must be below 0.05 per cent to eliminate aging effects.

The fact that welds made with covered electrodes, containing up to 0.08 per cent oxygen, are not hot short, whereas an oxidized steel containing this amount of oxygen is hot short, cannot be explained on the basis of total oxygen content. However, when the results of the fractional oxygen analysis are considered an explanation is found.

Figure 1 shows fractional oxygen analyses of the welds and plate material for which the chemical analyses are given in Table II. The total nitrogen contents of these three samples are also shown in this figure. It is noted that nearly all of the oxygen in the bare-wire weld appears in the first fraction as FeO and MnO. It has been observed in analyzing this type of weld metal, that the gas in the first fraction comes off rapidly which, according to Reeve,[8] indicates that it is largely FeO. Computations also show that most of the oxygen must be combined with iron. This particular weld had 0.09 per cent manganese, and, if all the manganese were in the form of MnO, it would combine with less than 0.03 per cent of oxygen leaving over 0.25 per cent oxygen as FeO. Referring again to Fig. 1, it is seen that most of the oxygen in the weld made with the covered rod is in the form of SiO_2, and only a small amount is in the form of FeO and MnO. From the rate of gas evolution during the analysis it is assumed that most of even this small amount is MnO. The data from which this chart was drawn show that the specimen contained 0.058 per cent oxygen combined with silicon as SiO_2 and 0.005 per cent oxygen combined with Mn, assuming that all of the FeO-MnO fraction is MnO. This weld analyzed 0.57 per cent manganese and 0.25 per cent silicon as shown in Table II. Deducting the manganese combined with the oxygen (0.017 per cent Mn) from total manganese and the silicon combined with oxygen (0.05 per cent Si) from total silicon, there still remains 0.55 per cent manganese and 0.20 per cent silicon in the metallic form which corresponds to

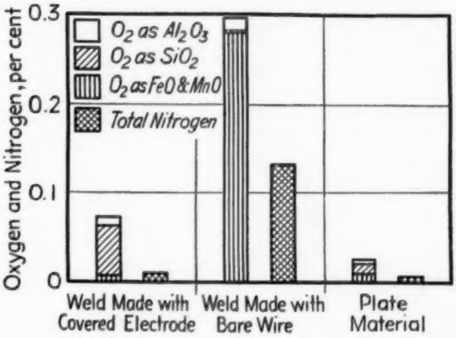

FIG. 1.—Chart Showing Nitrogen and Fractional Oxygen in Welds and Plate.

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[6] S. L. Hoyt, "Studies on the Metallurgy of Arc Deposited Weld Metal," *Transactions*, Am. Soc. Metals, Vol. 23, p. 61 (1935).

[7] F. R. Hensel and E. I. Larsen, "Age Hardening Phenomena in Typical Fusion Weld Metal," *Transactions* Am. Soc. Steel Treating, Vol. 19, p. 639 (1932).

[8] Lewis Reeve, "Improvements in Vacuum Fusion Method for Determination of Gases in Metals," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 113, p. 92 (1934).

a well-killed steel. When the fractional oxygen analysis of the weld metal made with covered electrodes is compared with that of the plate material, it is seen that the weld metal has approximately one-half as much oxygen in the first fraction as the plate. Since iron oxide is the harmful form of oxygen, it is only logical that the weld should show no ill effects due to oxygen.

The results of chemical analysis including fractional vacuum fusion indicate the importance of weld-rod coverings in producing weld metal with suitable properties. In welding with bare electrodes the desirable elements, carbon and manganese, are lost and the deposited metal absorbs large amounts of the harmful elements, oxygen and nitrogen. With properly covered electrodes the loss of carbon and manganese is minimized and objectionable contamination by oxygen and nitrogen is prevented. Other types of coatings such as dusted, washed, and lightly dipped improve the weld metal in proportion to the amount of protection furnished by the coating.

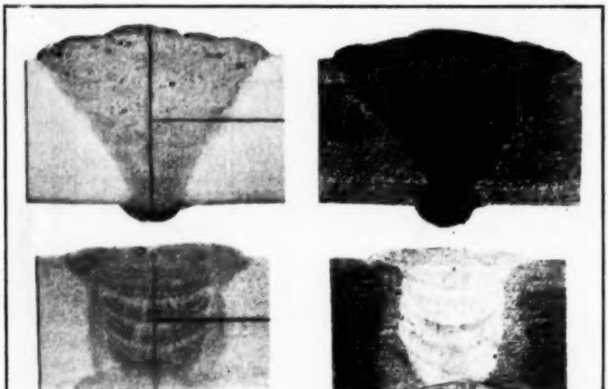

FIG. 2.—Cross-Section of Weld in 1-in. Plate (× 1).

Upper left—bare wire etched. *Upper right*—bare wire deep etched.
Lower left—covered electrode etched. *Lower right*—covered electrode deep etched.

Metallurgical Studies:

The results of metallurgical studies on weld metal have served to verify the findings of chemical analyses and to explain the behavior of various types of weld metal. In Fig. 2 are shown sections through welds made with bare and with covered electrodes. The weld made with bare wire had the following chemical analyses: carbon 0.01 per cent, manganese 0.09 per cent, silicon 0.01 per cent and nitrogen 0.139 per cent. This weld was made in the same manner as that for which the analyses are given in the first column of Table II and it will be noted that the two analyses correspond very closely. The mechanical properties of the weld shown in Fig. 2 were as follows:

Yield point, lb. per sq. in.	41 600
Tensile strength, lb. per sq. in.	43 900
Elongation in 2 in., per cent	1.0
Reduction of area, per cent	0
Bend elongation, per cent	1.0
Charpy value, ft-lb	1.6

The weld made with covered electrode and shown in Fig. 2 is the weld for which the analyses and mechanical properties are given in Table II (second column).

Some of the larger voids in the weld made with bare wire are visible in the lightly etched specimen. The deep-etched specimens show a marked contrast in the darkness of the print which is due to the amount of attack by the etching solution. Although the deep etch is not a corrosion test, it has been found from experience that weld metal which is severly attacked in the deep etch test often fails by corrosion in service, and weld metal which shows very little attack in the deep etch test is also resistant to corrosion in service.

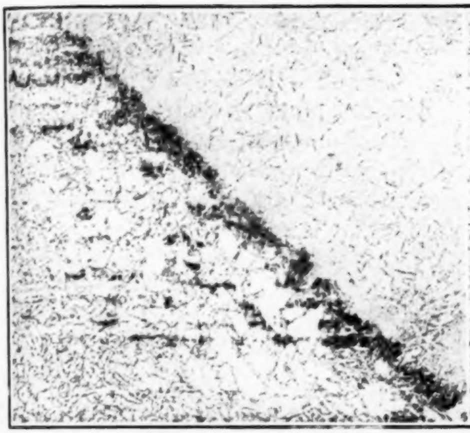

FIG. 3.—Junction of Weld Made with Bare Wire and Plate (× 100).
Stress relieved at 1200 F. Etched with 2 per cent Nital.

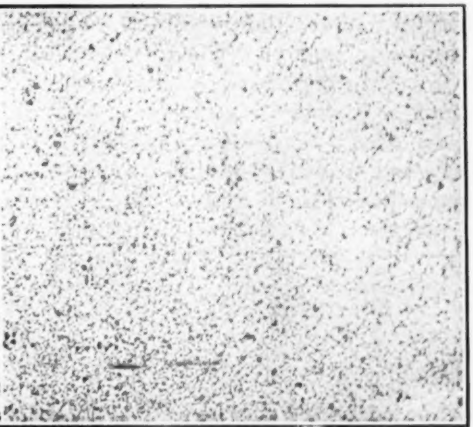

FIG. 4.—Junction of Weld Made with Covered Electrode and Plate (× 100).
Stress relieved at 1200 F. Etched with 2 per cent Nital.

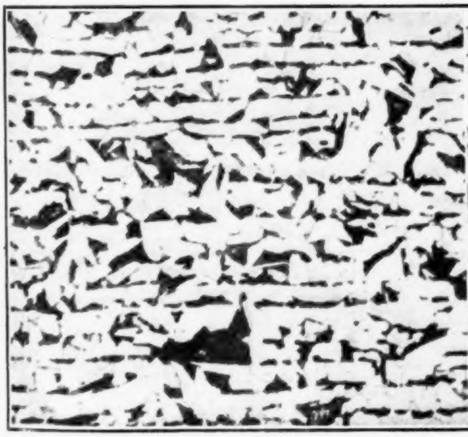

FIG. 5.—Unaffected Plate Stock (× 100).
Etched with 2 per cent Nital.

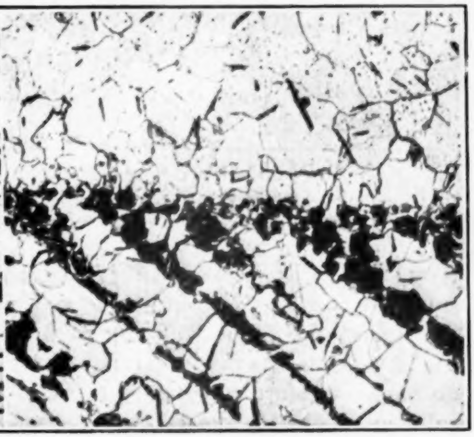

FIG. 6.—Junction of Weld Made with Bare Wire and Plate (× 200).
Annealed at 1700 F. Etched with 2 per cent Nital.

Further information concerning these two weld metals is obtained from a study of these specimens at a higher magnification. Figure 3 shows the junction or fusion zone of weld made with bare wire at 100 diameters and Fig. 4 is a similar photomicrograph of the weld made with the covered electrode. One difference between the two types of welding is very apparent. The demarcation between the deposited metal and the plate material in Fig. 3 is very sharp whereas the junction between the weld and plate stock in Fig. 4 is scarcely visible. Both the weld and stock in Fig. 4 show a highly refined grain structure resulting from the heat of welding. The structure of the plate before welding is shown in Fig. 5 which was taken beyond the zone affected by the welding heat. The original banded structure in the stock persists to a slight extent after the grain is refined by the welding operation resulting in the faint horizontal lines which distinguish the plate from the weld in Fig. 4. When the plate material shows no banding before welding, it is practically impossible to detect the junction of weld and stock after welding with covered electrodes. With bare electrodes the heat of welding is not sufficient to completely refine the plate material as is evident in Fig. 3.

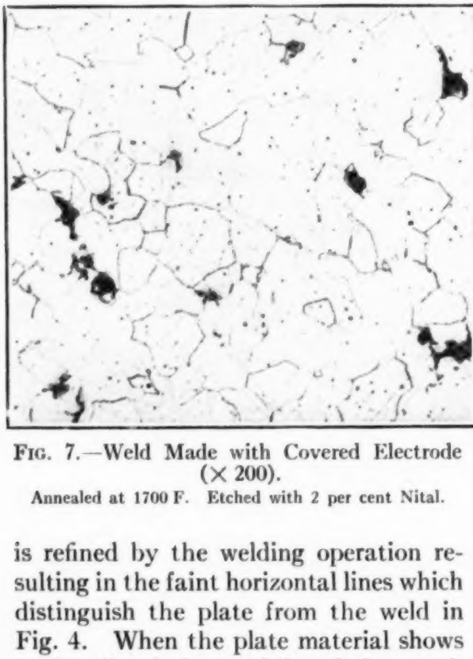

FIG. 7.—Weld Made with Covered Electrode (× 200).
Annealed at 1700 F. Etched with 2 per cent Nital.

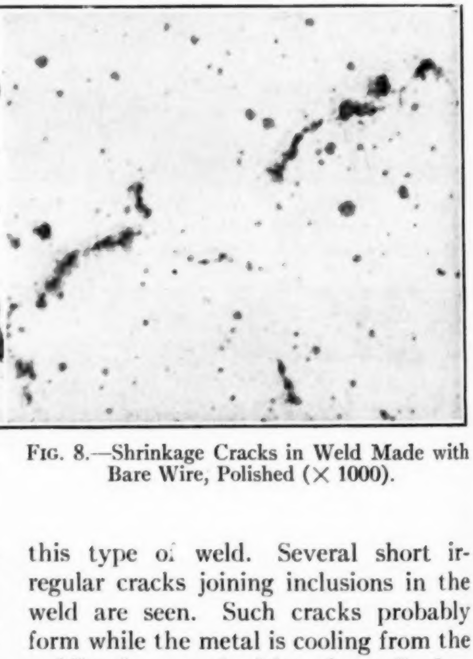

FIG. 8.—Shrinkage Cracks in Weld Made with Bare Wire, Polished (× 1000).

A careful examination of Fig. 3 shows fine nitride needles scattered throughout the weld. To bring these out more clearly a specimen of this weld metal was given a nitride anneal by heating to 1700 F. and furnace cooling. In Fig. 6, which shows this specimen at 200 diameters, the nitride needles are clearly visible. For comparison, Fig. 7 shows a specimen of the weld metal made with the covered electrode after the same heat treatment. These results are in agreement with the chemical analyses for nitrogen on these two types of welds.

A view at 1000 diameters of a polished specimen of a weld made with bare wire, shown in Fig. 8, offers a possible explanation for the erratic strength values of this type of weld. Several short irregular cracks joining inclusions in the weld are seen. Such cracks probably form while the metal is cooling from the welding heat, and, although small, they act as nuclei of failure when the weld is stressed, resulting in the low strength values observed for some specimens.

For the identification of inclusions in metals, Hoyt and Scheil[9] have shown that reflected polarized light is very helpful. Using this method Hoyt[6] has

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[9] S. L. Hoyt and M. A. Scheil, "Use of Reflected Polarized Light in Study of Inclusions in Metals," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 116, p. 405 (1935).

shown that the inclusions in welds made with bare wire have a high FeO content and welds made with covered electrodes have very little FeO. These findings agree with the results of the fractional vacuum fusion analysis.

Correlation of Results of Chemical Analyses and Metallurgical Examination with Mechanical Properties:

The results of metallurgical studies of weld metal are confirmed by chemical analyses including vacuum fusion results. As a result of both methods of investigation it has been found that weld metal made with bare wire contains large amounts of iron oxide and nitrogen whereas weld metal made with covered electrodes contains relatively small amounts of these. Results of physical tests of steels containing excessive amounts of iron oxide and nitrogen indicate that the lack of ductility and toughness of welds made with bare electrodes are due to the high iron oxide and nitrogen content of this type of weld metal.

RESULTS OF WELDING ALLOY STEELS

During the past few years there has been an increasing demand for the use of welding in fabricating structures of alloy steels of both the low-alloy and the corrosion-resistant types. The reported data on alloy welds are meager compared with the data on carbon-steel welds because much more welding has been done on carbon steel. With the background obtained from studies of welds in carbon steel it is not necessary to make such an extensive investigation of alloy welds. For most purposes, physical tests, routine chemical analyses, and in some cases corrosion tests are sufficient.

The mechanical properties of weld metal suitable for welding some of the more commonly used alloy steels are given in Table III. For the carbon-molybdenum, nickel, and chromium-vanadium steels, $\frac{1}{2}$-in. round all-weld metal specimens were used. For the three chromium steels flat tension specimens along the weld were tested because these materials were from $\frac{1}{4}$ to $\frac{1}{2}$ in. thick and the $\frac{1}{2}$-in. round specimens could not be obtained.

These results are average values. From ten to twenty specimens of each type of weld were tested except the stainless 18 per cent chromium, 8 per cent nickel alloy which had six and the 4 to 6 per cent chromium alloy which had three tension specimens. In addition to the tension specimens of the 4 to 6 per cent chromium material, several bend specimens were tested and these all showed elongation of over 30 per cent. Also, tension specimens across the weld developed the strength of the plate material.

TABLE III.—MECHANICAL PROPERTIES OF WELD METAL FOR SOME ALLOY STEELS.

Kind of Steel	Yield Point, lb. per sq. in.	Tensile Strength, lb. per sq. in.	Elongation in 2 in., per cent
Carbon-molybdenum (Mo 0.40 to 0.60%)	57 200	72 900	27.9
Nickel (Ni 2.0%)	60 900	74 400	25.6
Chrom-vanadium (Cr 1.0%, V 0.20%)	74 800	91 100	18.5
Chromium (Cr 4.0 to 6.0%, Mo 0.40 to 0.60%)	42 300	77 300	30.8
Stainless (Cr 16 to 18%)	58 700	85 000	22.5
Stainless (Cr 18%, Ni 8%)	46 500	75 200	22.5

The welds in the carbon-molybdenum, nickel, and chromium-vanadium steels were all stress relieved at 1200 F. The 4 to 6 per cent chromium specimens were annealed at 1600 F. and the 16 to 18 per cent chromium specimens at 1400 F. The 18 per cent chromium, 8 per cent nickel specimens were not annealed.

In addition to these materials there

have also been developed recently a number of low-alloy, high-tensile strength steels which are designed primarily for welding. In these steels the strength is obtained from such elements as copper, nickel, molybdenum, chromium, and manganese while the carbon is kept low to prevent air hardening. Gibson[10] shows the strengths obtained in welding some of these alloys. From his results it appears that yield points of the welds are as high or higher than those of the plate material, and many of the specimens failed in the parent metal, indicating that the tensile strengths of the welds are ample.

Although the number of tests on alloy welds are not numerous compared with those on carbon steel, the available data indicate that many of the alloy steels can be just as successfully welded as carbon steel.

SUMMARY

The status of weld metal as an engineering material has changed greatly during the past fifteen years. Although the weight of the weld compared to the weight of the other material used in any structure is almost insignificant, the quality of the weld metal is just as vital to the success and safety of the structure as the quality of the material welded. For the determination of the properties of weld metal it has been necessary to supplement routine testing methods with special physical tests, chemical analyses, and metallurgical examinations. As a result of these investigations, the behaviour of weld metal has been explained and the causes for inferior quality in welds have been determined. These studies have thus established that the quality of weld metal is not dependent upon some mysterious circumstances but is the result of welding technique and procedures which can be controlled. Because of the reliability and the high quality of weld metal obtainable by modern procedures, weld metal has assumed an important place among engineering materials.

Acknowledgment:

The author wishes to acknowledge the cooperation and helpful criticism of his colleagues in the A. O. Smith Corp. and to thank the officers of the A. O. Smith Corp. for permission to publish these results.

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[10] A. E. Gibson, "The Development and Use of Low Alloy High Tensile Steels," *Journal*, Am. Welding Soc., Vol. 14, p. 2 (1935).

DISCUSSION

MR. J. J. CROWE[1].—I should like to point out one thing in connection with Mr. Larson's paper. He speaks about the physical characteristics of the different specimens consisting entirely of weld metal and infers that these characteristics are a function of the welding rod and are independent of the metal being welded. If you take a given welding rod and make a weld between two plates the physical characteristics of the weld metal in gas welding and, I believe, to a certain extent, in covered electrode welding, depends largely on the composition of the plates being welded. For instance, if you weld two plates having a tensile strength of 60,000 lb. per sq. in. you are likely to get approximately 60,000 lb. per sq. in. tensile strength in the weld metal. If, however, using the same welding rod, you weld two plates having a tensile strength of 80,000 lb. per sq. in., the tensile strength of the deposited weld metal will be appreciably higher than 60,000 lb. per sq. in. and may be as high as 80,000 lb. per sq. in.

The diffusion taking place during welding is such that the physical characteristics of the weld metal are greatly influenced by the chemical composition of the metal being welded.

MR. L. J. LARSON[2] (*author's closure, by letter*).—I agree with Mr. Crowe that the properties of the weld metal may be affected by the composition of the metal being welded. The amount of alloy added to the weld metal by the fusion of the base material depends upon the amount of base material fused and upon the composition of the base material. The amount of base material fused depends upon the welding process and upon the procedure used. With gas welding the weld metal may consist largely of molten base material in which case the mechanical properties of the weld metal will be affected to a great extent by the composition of the base material. On the other hand with multi-pass arc welding in heavy plates a relatively small amount of base metal is melted and hence the composition of the base material has much less effect on properties of the finished weld.

Many of the covered electrodes used for welding mild steel having a minimum tensile strength of 55,000 lb. per sq. in. produce weld metal having a tensile strength of 65,000 to 70,000 lb. per sq. in. Although the tensile strength of the weld metal produced by using these same electrodes in welding high-strength steels will be higher, it is common practice to use other types of electrodes for welding steels having a minimum tensile strength of 70,000 lb. per sq. in. and still other electrodes if a tensile strength of 85,000 to 100,000 lb. per sq. in. is required. Using mild steel plates, with different electrodes it is possible to produce, at will, weld metal having tensile strengths ranging from 55,000 to 100,000 lb. per sq. in.

For welding thin sections of low-alloy steels which are not stress relieved after welding, electrodes used for mild steel may produce weld metal having sufficient tensile strength, but for welding heavy plates which are stress relieved it is necessary to use different electrodes for steels of different tensile strengths. The alloys obtained from the fusion of the base material are not sufficient to adjust automatically the strength of the weld metal to the strength of the material being welded.

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[1] Manager, Apparatus Research and Development Dept., Air Reduction Sales Co., Jersey City, N. J.

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STEEL STRUCTURES IDENTIFIED AND FLAWS LOCATED BY MEANS OF BALANCING WAVE TESTS

By Carl Kinsley[1]

Synopsis

Non-destructive tests of the uniformity of steel samples or of the character and amount of their difference are made by electromagnetic measurements with a dynamometer and subsidiary phase shifting transformer.

Before a sample has been treated so as to change its structure it is placed in the magnetizing coil and the electromotive force induced in the testing circuit is measured with respect to an empty coil—an *air* core.

This initial electromotive force is completely balanced by introducing into the circuit an equal, but opposing electromotive force. This is synthesized from the phase shifting transformers which term by term neutralize the induced electromotive force. The opposing electromotive force is called the *balancing wave* and it has a sign opposite to that of the corresponding induced electromotive force.

Each sample is now treated so as to change its structure. It is then placed in the magnetizing coil and the value of the electromotive force is obtained as a series of harmonic terms. The amplitude and phase angle of each of the significant terms of the complete wave is evaluated and their interpretation gives the character and the amount of the change produced in the sample.

Illustrations are given of the metallurgical differences between the samples and between different sections of the same sample, also, of the effect of different tempering temperatures on similar samples which had been quenched alike, with an error in one treatment indicated, and finally, of the location of a crack in one of a series of rods which otherwise differed only within allowable limits.

Non-destructive methods of determining the structure of metals or the presence of flaws are extensively employed. Among them might be included the methods using X-rays, gamma rays, magnetic quantities, sonic and supersonic waves, heat development and its rate of diffusion, together with a considerable number of tests collectively known as magnetic analysis.[2]

Electromagnetic measurements are employed in the last named type of test, and they have been successful in a limited field of operations. The limitation is due to factors of minor importance with respect to the material being tested, but which sometimes render the correct interpretation of the measurements impossible and always leave a feeling of uncertainty with regard to the results. Therefore, a method of testing which makes it possible to interpret the results correctly is essential.

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[1] Consulting Engineer, New York City.

[2] See bibliographies by R. L. Sanford appearing in *Journal*, Am. Inst. Electrical Engrs., Vol. 48, June, 1929, pp. 7–11; *Transactions*, Am. Soc. Steel Treating, Vol. 5, p. 577 (1924).

In the method herein described,[3] there is introduced into the circuit a balancing wave of electromotive force, composed of the controlled output of phase shifting transformers, which can completely neutralize any or all of the components of the complex electromotive force in the testing circuit. By balancing the effect of the disturbing factors, it is possible to make correct interpretation of the remaining electromotive force in terms of the structure and physical properties of the material.

Many applications of this method of testing have been made in manufacturing plants and associated laboratories. From the results obtained, three typical tests have been chosen as illustrations.

THEORY OF THE METHOD OF TESTING

Fundamental Assumption:

It is believed that the hysteresis loops uniquely characterize any ferromagnetic material with respect to its structure and physical condition. The magnetic "finger print" is standardized and recorded by using a simple sine wave of magnetizing force which produces a cyclical magnetic flux in the material which, in turn, causes a complex induced electromotive force in a secondary testing circuit. This is quantitatively analyzed by the testing apparatus into its equivalent Fourier's series of harmonic terms which are then recorded as the complete characterization of the material. It has not been found possible to make any change in the material without having a corresponding change in one or more of the constants of its characteristic series of terms. No different combination of analysis, metallurgical structure or physical condition has been found that would produce a duplicate of the characteristic equation of the specimen.

Equations Defining the Test:

The magnetizing current, which can be taken from a Public Service power line or from any constant-frequency a.c. generator having a line filter to insure its sine wave form, can be represented by the equation,

$$i = I \sin\omega t \;\ldots\ldots\ldots\ldots (1)$$

The complex induced electromotive force in the testing circuit, which depends on the shape of the hysteresis loop, can be represented by the equation,

$$\begin{aligned} e = {} & V_1 \sin(\omega t - \phi_1) + V_2 \sin(2\omega t - \phi_2) \\ & + V_3 \sin(3\omega t - \phi_3) + V_4 \sin(4\omega t - \phi_4) \\ & + \cdots V_n \sin(n\omega t - \phi_n) \ldots\ldots\ldots (2) \end{aligned}$$

where i = the value of the current at the time t,

I = the maximum value of the current,

ω = the angular velocity of the current vector, $\omega = 2\pi/T$,

T = the time in seconds of a complete cycle,

t = the time from the start of the current wave, chosen as the zero,

e = the value of the complex electromotive force at the time t (when a specimen is used a subscript is added),

V = the maximum value of each of the harmonic terms in electromotive force units, the harmonic used is shown by the subscript,

ϕ = the phase of the harmonics, or the angle in radians by which the harmonic leads or lags with respect to the zero of the current wave.

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[3] United States Patents No. 1,743,087 (1930), and No. 1,910,770 (1933).

It has been found that usually only a limited number of these terms need be used; also for symmetrical magnetization only the odd terms are present.

The complete method of measurement is suitable for the most exacting laboratory investigation and also for determining what approximations can be used in commercial mill operations to give dependable results at the high speeds necessary for commercial purposes.

FIG. 1.—Specimen B Alone and with Its Balancing Wave.

FIG. 2.—Specimen F_1 with the Balancing Wave of Specimen B.

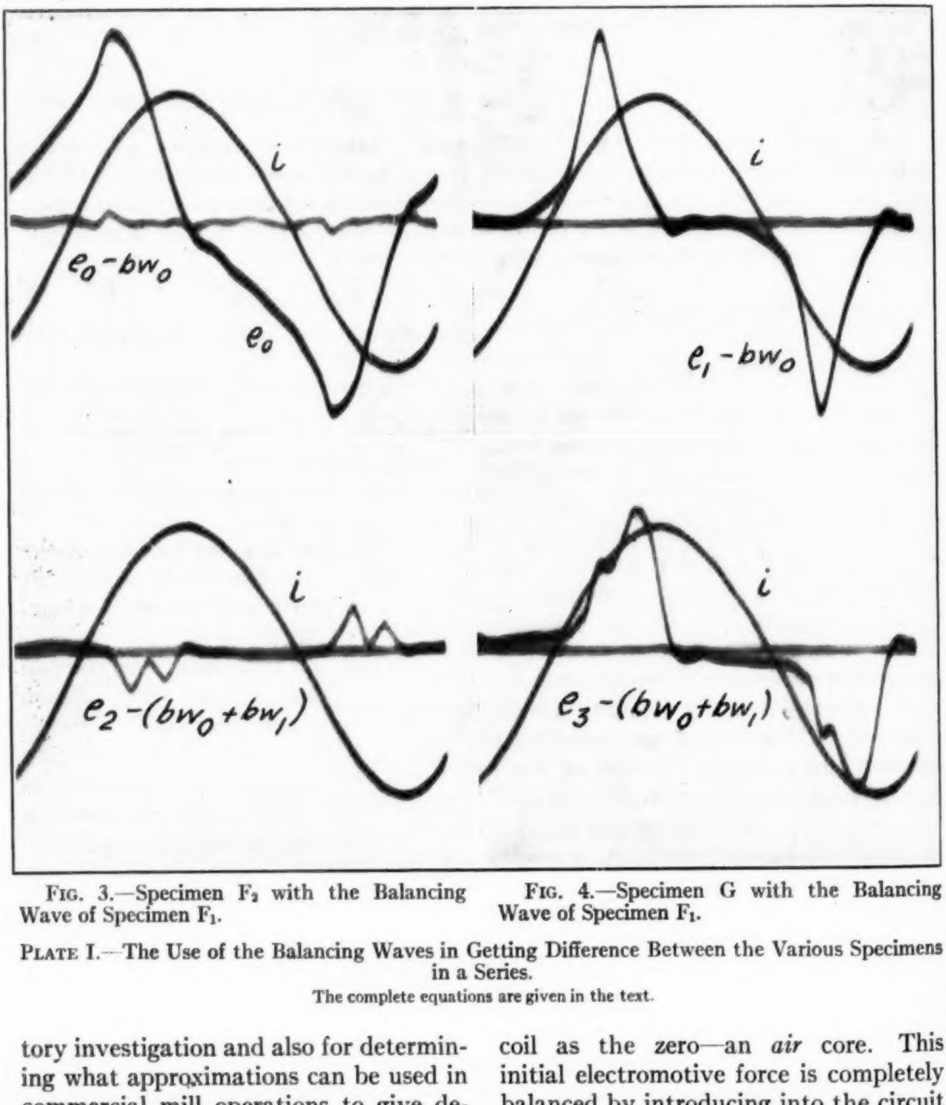

FIG. 3.—Specimen F_2 with the Balancing Wave of Specimen F_1.

FIG. 4.—Specimen G with the Balancing Wave of Specimen F_1.

PLATE I.—The Use of the Balancing Waves in Getting Difference Between the Various Specimens in a Series.

The complete equations are given in the text.

Laboratory methods of measurement will be considered first.

The electromotive force, e_0, induced in the circuit when one of the specimens to be used in a series is present in the coil, is measured with respect to an empty coil as the zero—an *air* core. This initial electromotive force is completely balanced by introducing into the circuit an equal, but opposing, electromotive force. This is synthesized from the phase-shifting transformers which term by term neutralize the induced electromotive force. The opposing electromotive force is called the *balancing wave*, bw, and it has a sign opposite to that of the corresponding induced electromotive force e_0. The electromotive force equation becomes:

$$e_0 = bw_0 \text{ or } e_0 - bw_0 = 0 \ldots\ldots (3)$$

It is essential for any exact experimental work that all of the specimens of a series, which are to be subjected to any treatment, shall be substantially alike. They should, therefore, be measured and their characteristic equations recorded. It is not sufficient that they be cut from a single rod but they must differ by an amount no more than that known to be allowable for the particular purpose for which they are to be used.

It is assumed that any treatment causing a change in the steel specimen produces an additional electromotive force, e_1, over and above that neutralized by the balancing wave, bw_0. The change in the steel is then correlated with the corresponding electromotive force, e_1. If a further treatment is to be given to the steel specimen, a new point of departure is obtained by adding to the circuit a balancing wave, bw_1, which reestablishes the zero by neutralizing e_1.

The balancing waves can be combined into one by their vector addition and the equation becomes,

$$e = e_0 + e_1 = bw_0 + bw_1 = bw_{01} \ldots (4)$$

$$\text{or } e - bw_{01} = 0$$

The next step in the treatment causes a further additional electromotive force, e_2, which is correlated with the second change in the steel.

The use of two balancing waves is not necessary. There is a practical advantage, however, in increased accuracy. When the total electromotive force to be measured is small, the zero should be fixed with precision. This is easily done with the extra balancing wave, bw_1.

The above process is illustrated by measurements made on specimens of rods intended for a manufacturing purpose requiring the material to have high ductility.[4] Oscillograph records illustrating the electromotive forces in the testing circuit are reproduced in Figs. 1 to 4.

Figure 1 shows by an oscillograph record the total electromotive force induced in the testing circuit when specimen B is used alone. The zero is therefore given by the empty magnetizing coil—by *air* alone. Also, after the balancing wave is introduced the electromotive force becomes zero, except for harmonics above the seventh. This electromotive force has only the odd harmonics present as the positive and negative portions of the curves are similar. The amplitude and phase of each of the terms of the first, third, fifth and seventh harmonics of Eq. 2 are given in the following equation:

$$e_0 = 57.0\underline{/-67^\circ} + 14.1\underline{/+15^\circ} - 3.4\underline{/-10^\circ} - 0.3\underline{/+50^\circ} \ldots (5)$$

Figure 2 illustrates the change in the electromotive force induced in the testing circuit when specimen B is replaced by specimen F_1. This is measured from the second zero by retaining the balancing wave of specimen B in the circuit, as follows:

$$e_1 = 34.0\underline{/-45^\circ} + 18.7\underline{/+32^\circ} - 8.1\underline{/-16^\circ} - 4.0\underline{/+85^\circ} \ldots (6)$$

The total electromotive force is now the algebraic sum of the two series taken

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[4] The specification requirements of the material in question are given under Data and Discussion; photomicrographs are shown in Fig. 7.

term by term and added vectorially so that another balancing wave, bw_1, can be added to the circuit so as to "clear the deck" in preparation for another change in the specimen, as shown by Eq. 4.

Figure 3 shows a relatively small change in the electromotive force when specimen F_2 is substituted for specimen F_1.

$$e_2 = -4.3\underline{/-9^\circ} - 4.9\underline{/+29^\circ} + 2.8\underline{/-36^\circ} + 2.3\underline{/+45^\circ} \ldots\ldots (7)$$

The two specimens F_1 and F_2 show test results that are relatively close, as shown on the graph of Fig. 5, and they would be expected to have closely related structure.

Figure 4 shows the circuit in a balanced condition for specimen F_1 but with specimen G in the testing coil. The specimens are thus compared through their common zero. The difference between specimen F_1 and specimen G is then given by the following equation:

$$e_3 = 31.2\underline{/-34^\circ} + 16.6\underline{/+86^\circ} + 6.9\underline{/-72^\circ} - 3.8\underline{/-47^\circ} \ldots\ldots (8)$$

NOTE.—This equation shows the characteristic phase values of the first term which accompanies the structure change seen in the micrographs given in Fig. 7.

It may be pointed out here that this method is suitable for determining very small changes in structure. For instance, it has been used to follow the progressive structural changes due to aging. At first the time interval between records is made very short as the electromotive force changes rapidly.

That it is a function of the structural change of the steel due to aging seems probable. The electromotive force values for carbon aging, when plotted, give a smooth curve with a well-marked transition point at its maximum value. It is generally accepted that aging is caused by structural changes, although its observation "under the microscope is not very successful." Physical tests, such as Rockwell hardness measurements, can follow such changes and give a rough value of the trend. Carbon aging causes a maximum hardness and a slow approach to equilibrium.

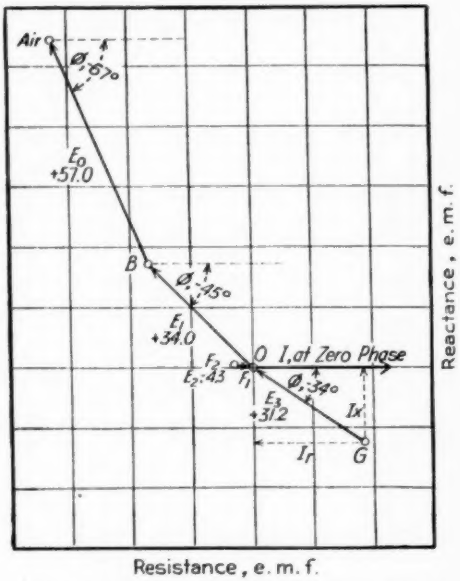

FIG. 5.—Electromotive Forces Plotted, Using Polar Coordinates.

Resistance and reactance components or their vector sums can be obtained by inspection using the Cartesian coordinates.

When the aging change is plotted against the time, the curve will be wholly consistent in each case irrespective of the initial variations between the specimens. This method of measurement has produced complete and conclusive results.

SIMPLIFICATION AND INTERPRETATION OF THE EQUATIONS

The fundamental term of the characteristic series is particularly significant since it alone carries the equivalent resistance component of the vector electromotive force (or the energy loss term, since the magnetizing current is constant) while the total reactance is the sum of the reactance values of all of the terms of the series.

The harmonic components above the first are essential for the solution of certain problems, but they can be omitted in many investigations. Very extensive laboratory tests have established the requirements for many practical cases in both laboratory and commercial work. For instance, the even harmonics are needed with permanent magnetic material or when the material is biased by a direct current component in the magnetizing current. Also, decarburized specimens can be tested and the decarburization located with the use of the seventh harmonic alone, even in the presence of large differences in analysis and in the structure of the base metal.

The use of a single term of the electromotive series is illustrated in Fig. 5, where only the first harmonic of the series expressing the waves given in Figs. 1, 2, 3, and 4 is employed, taken from Eqs. 5, 6, 7, and 8.

The interpretation of the results depends on the phase angle of the electromotive force with respect to the current and this is best understood from the relation between the quadrature components, consisting of the resistance electromotive force in phase with the current vector and the reactance electromotive force in quadrature with it. The following expressions give the relationships:

$$i = I_m \sin \omega t; \qquad e = E_m \sin (\omega t - \phi) \ldots\ldots (10)$$

These equations give the periodic curves of the current and electromotive forces.

For I located at $\phi = 0$,

$$E = Iz\underline{/-\phi} \ldots\ldots\ldots\ldots\ldots\ldots (11)$$

The current and electromotive force can be expressed in vector notation, using virtual values as they are read from the measuring instruments:

$$E = I\ (r - jx) \text{ with the phase, } \quad \phi = \tan^{-1} (-x/r) \ldots\ldots (12)$$

The resistance component of E is Ir and the reactance component of E is Ix.

The symbols represent the following quantities:

i, I_m = the instantaneous and maximum value, respectively, of the current sine wave,

e, E_m = the instantaneous and maximum value of the electromotive force wave,

I, E = virtual values of current and electromotive force, $I = 0.707\ I_m$, $E = 0.707\ E_m$, which are measured in amperes and volts by the usual ammeters and voltmeters,

r = the equivalent resistance so that I^2r is equal to the total loss in watts,

x = the reactance of the circuit, equal to $L\omega$. With constant frequency the magnetic flux in the specimen is proportional to L,

L = the self induction of the circuit,

ω = the angular velocity of the current vector or $= 2\pi/T$ or 2π multiplied by the number of cycles per second,

Ir = the resistance electromotive force which is the component of the electromotive force in phase with the current,

Ix = the reactance electromotive force in quadrature with the current,

Iz = the impedance multiplied by the current, or $E = Iz$.
$z = \sqrt{r^2 + x^2}$, and

$E_0, E_1, E_2, E_3,$ = The electromotive forces expressed as virtual voltages of the first terms of the series of harmonic terms. The subscripts refer to particular values measured when there were differences in the steel.

The current vector, I, and the additional electromotive force vector, E_3, are plotted in polar coordinates from the zero where specimen F_1 is balanced by $(bw_0 + bw_1)$. The current, I, is in the zero phase position and has a value of 0.055 amp. The electromotive force vector is at a phase angle of -34 deg. and the dynamometer deflection is 31.2 scale divisions, $31.2\underline{/-34 \text{ deg.}}$ The deflection is proportional to the electromotive force in volts. This quantity results from the structural differences between specimen F_1 and specimen G.

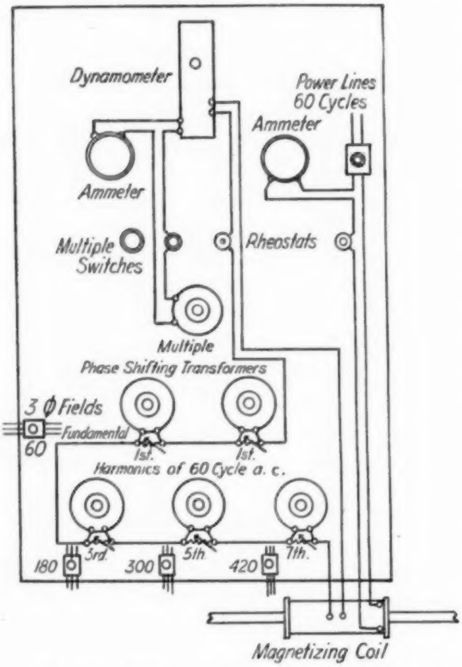

FIG. 6.—Schematic Diagram of Circuits for Balancing Wave Method of Testing.

The electromotive force, E_3, is resolved into components and shown on the graph in Cartesian coordinates. The resistance e.m.f., Ir, is parallel to the current axis, and the reactance e.m.f., Ix is in quadrature to it. The phase of E_3 with respect to the current vector, I, is $\tan^{-1}(-Ix/Ir)$ and is shown equal to -34 deg.

These relationships are of fundamental importance with respect to the character of the steel and its physical properties. Specimens F_1 and B were cut from sections of the same rod only 3 ft. apart. They are typical of the variations that often occur in rods of this material subjected to the same treatment. There is a progressive change from one culminating point to another and a continuous variation in the electromotive force and in the structure of the material. It can be stated positively, after many investigations of different types of structure, that there has been found no exception to the conclusion that *the change in the electromotive force, measured as described here, is a function of the change in the structure of the steel sample.*

TESTING APPARATUS

Figure 6 is a schematic diagram showing the essential elements of the equipment. The apparatus employed consists of the following:

1. A sensitive dynamometer for laboratory measurements.
2. Ammeters for laboratory work to maintain the magnetizing current constant and to check the field current of the dynamometers as the different harmonics are used.
3. Phase shifting transformers made from selsyn motors.
4. Magnetizing coil built as needed.
5. Generators of harmonic frequencies of which only a limited number are needed for any particular purpose. The output needed is ten watts per phase.
6. Magnetizing current from commercial power line.

7. Control resistances in the various circuits.

For commercial work the apparatus may be greatly simplified. The apparatus is set up to indicate when certain tolerance limits are exceeded. Precise measurements are not needed for this type of work nor can they be made at the high speed necessary in an operating mill.

The apparatus, for example, can be limited to a single indicating or recording instrument which is relatively insensitive and usually only a single selected harmonic phase-shifting transformer is needed to supply the required balancing wave. It is not necessary to readjust this unless there is a change in the base metal which places the zero outside of the tolerance circle previously used.

The operation of the balancing wave method consists of the following successive steps:

1. The magnetizing current and the current in the field of the dynamometer are adjusted to their constant value. (This is not required in commercial mill operations.)

2. The phase angles of the various harmonics used are determined from the dial readings of the phase shifting transformers.

3. The dynamometer is brought to zero, using a balancing wave compounded of all of the harmonics needed. Each is adjusted separately as the field of the dynamometer is energized in succession with a current of the same frequency, drawn from its phase-shifting transformers through the multiple switch, proceeding as follows:

(*a*) The dynamometer is brought to zero by putting the phase angle of the field current in quadrature with the effective component of the current in the moving coil.

(*b*) The phase angle of the field current is changed through 90 deg. so as to put the balancing wave in opposition to the component of the electromotive force of the same frequency.

(*c*) The amplitude of the balancing wave is changed so as to bring the dynamometer to zero. The dynamometer will remain at zero for any phase angle of the field current.

4. The amplitude and phase of the additional electro-electromotive force due to the differences between the samples is measured, each harmonic being obtained separately. The amplitude is given by dynamometer deflection and the phase is taken from the transformer dial reading when the deflection is a maximum.

DATA AND DISCUSSION

Identification of Structure:

An instance is given of a cold-drawn steel, treated so as to have high ductility. The material was drawn alloy steel, annealed and pickled, 0.183 in. by 0.053 in., intended to be wound on a mandrel of 0.90 in. in diameter. The chemical analysis was as follows:

Carbon, per cent	0.45 to 0.55
Chromium, per cent	0.85 to 1.10
Molybdenum, per cent	0.20 to 0.30
Nickel, per cent	1.75 to 2.25
Manganese, per cent	0.40 to 0.60
Silicon, per cent	0.15 to 0.30
Phosphorus and sulfur, per cent	0.04 to 0.00

A set of three samples, duplicating the one used in making the electromagnetic measurements shown in Figs. 1 to 4, and a second similar set were independently selected. The first was sent to the New York Testing Laboratories and the second was sent to the National Physical Laboratory, Teddington, England.

The reports from the two laboratories checked completely and confirmed an extensive series of similar tests and examinations made previously in the course of the work. The report of the National Physical Laboratory follows:

REPORT OF THE NATIONAL PHYSICAL LABORATORY, TEDDINGTON

Longitudinal sections cut through each of the marked areas have been prepared and etched in a 4 per cent solution of nitric acid in alcohol. Figs. 1, 2, and 3[5] show the microstructures at 1000 diameters of the three specimens G, F and B, respectively. In G the carbon is present in the form of spheroidized cementite and the grain size of the material is very fine. In specimen F, Fig. 2, the carbon is not completely spheroidized and is partly present as pearlite. In the material B the carbon for the most part is in the form of pearlite, but a proportion of it is present as sorbite. The grain size of specimen B is coarser than either that of G or F.

Conclusions.—The difference in the microstructure of the three areas has been caused by a difference in the final heat treatment of the material. The heat treatment of material G has resulted in the separation of carbide as granular cementite, but that of material B has resulted in the formation of masses of pearlite and sorbite, and has caused a reduction in the ductility of the steel. The spheroidization of the cementite of material F is not complete and the mechanical properties of this material lie, as would be expected, between those of G and B.

(signed) WALTER ROSENHAIN

The photomicrographs made by the National Physical Laboratory are reproduced in Figs. 7 (*a*), (*b*), and (*c*). Notched bar tests with the Izod machine made at the same laboratory gave the following results, upon which the conclusions with respect to ductility are based:

	ENERGY TO FRACTURE
Specimen G	0.80 ft-lb.
Specimen F	0.75 ft-lb.
Specimen B	0.26 ft-lb.

In the course of the investigations preceding the above laboratory measurements, hundreds of specimens were examined by electromagnetic measurements and the specimens were then tested by winding them on their edge on the mandrel in the regular production operations in the mill. It was found that the metal of low ductility was invariably detected while no metal of sufficient ductility was incorrectly indicated.

The data from the oscillograph records and the electromagnetic measurements of the above steel are given in Figs. 1 to 5.

Applications:

The balancing wave method of testing is applicable both to exact research investigations and as a reliable means of inspection for commercial purposes.

Research Applications.—With the balancing wave method of measurement it is possible to make complete records of complex electromotive forces, to establish the equality of samples at any time, to identify them and to repeat or to extend the examinations of the samples as may be needed, since the measurements are all non-destructive of the material. Figures 1 to 5 illustrate how the records are made and a continual check maintained on the samples. The electromotive force differences between the samples can be determined to 0.1 per cent of the electromotive force of the samples.

The structural difference between the specimens F_1 and F_2, Fig. 5, are too slight to be shown by photomicrographs. But the structural differences between specimens G, F, and B, on Fig. 7, are indicated by the curve in Fig. 5. The relative position was confirmed by the physical test values. The illustration cited is typical of hundreds of determinations as made by photomicrographs and accompanying electromotive force readings. The progressive changes in the structure of the steel are directly reflected by the corresponding changes in the electromotive force values as measured by the balanced wave method.

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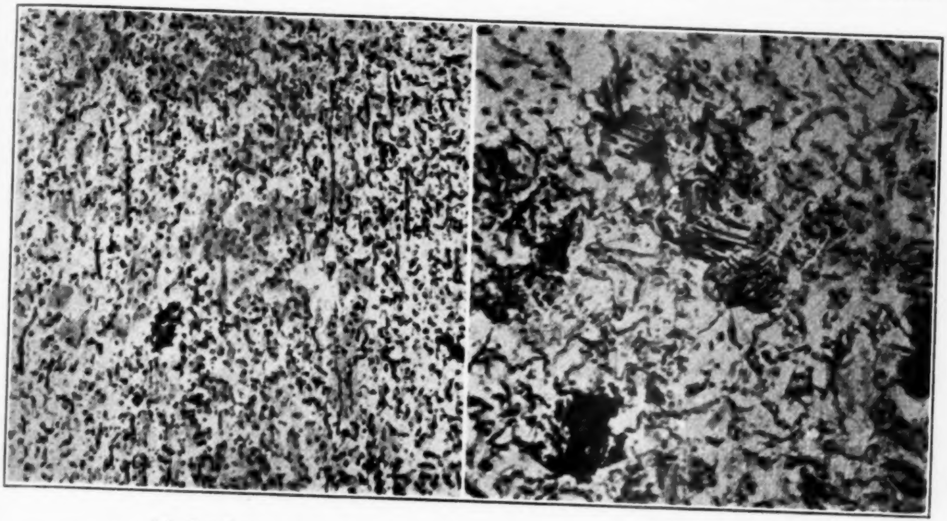
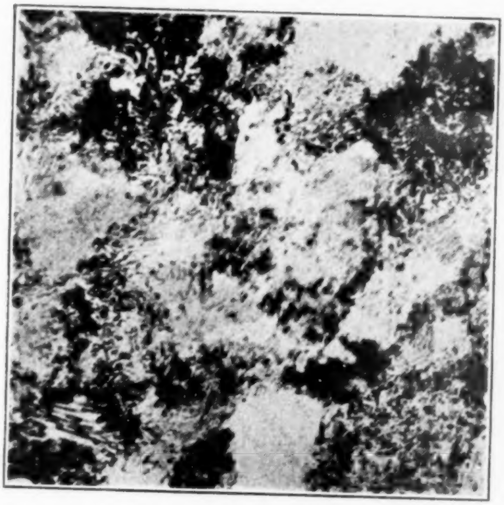
[5] The accompanying Figs. 7 (*a*), (*b*) and (*c*).



(*a*) Specimen G.

(*b*) Specimen F.



(*c*) Specimen B.

FIG. 7—Photomicrographs of Specimens of Cold-Drawn Rods Showing Progressive Changes of Structure Accompanying Reduction in Ductility (× 1000).

All specimens are etched in a 4 per cent solution of nitric acid in alcohol.

Commercial Applications.—In the commercial use of the apparatus the balancing wave can be fixed at such a pre-established value with reference to the material that the appearance of a particular additional electromotive force will indicate the presence of an undesirable structure. This can be readily done in many cases by the proper adjustment of the balancing wave since it can cause the additional electromotive force to differ from the one normally present only by a phase change or by an amplitude change alone or by the two in combination. In this case the balancing wave is not started with the electromotive force reduced to zero but rather to a value which will allow the selective operation of the relay to be easily controlled.

As applied to ductility measurements, the test has been used in mill operations, and in the testing of 50,000 ft. of intermittently imperfect material, all of the material with ductility too low was invariably located by the electromagnetic tests. The dividing line was established by preliminary tests and fixed at a value which placed it between specimen F_1 and specimen B shown in Fig. 5.

An example of the commercial use of the test is that in connection with the tempering of alloy steel rods to obtain the structures having specific physical properties. The rods were of alloy steel, 0.5625 in. in diameter and 18.4 in. in length, which had been quenched in oil at 850 C. and were used for making studs for airplane engines by one of the leading manufacturers in England.

The chemical analysis was as follows:

Carbon, per cent	0.22 to 0.27
Chromium, per cent	1.10 to 1.40
Molybdenum, per cent	0.50 to 0.00
Nickel, per cent	not specified
Manganese, per cent	0.40 to 0.65
Silicon, per cent	0.30 to 0.00
Phosphorus and sulfur, per cent	0.03 to 0.00
Tungsten, per cent	2.75 to 3.25
Vanadium, per cent	0.25 to 0.00

The required physical properties were as follows:

Tensile strength, lb. per sq. in.	156 800 to 168 000
Elongation in 2 in., per cent	17
Reduction of area, per cent	40
Izod test, ft-lb	50
Brinell hardness, minimum	331

The heat treatment to produce these physical properties comprised oil hardening at a temperature of 850 C. followed by tempering at between 560 and 630 C. the particular temperature depending on the actual chemical composition within the specified limits. The range of interest is, therefore, that lying at the lower end of the curve in Fig. 8. An extensive series of experiments was carried out for the temperatures between 500 and 650 C.

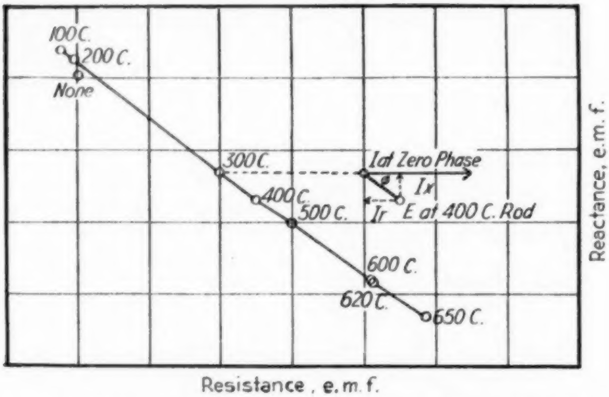


FIG. 8.—Electromotive Force Values Plotted in Polar Coordinates.
Quenched at 850 C. Each rod tempered as shown.

The data for the electromagnetic measurements are given in Fig. 8. The values in every case gave the same progressive changes as the tempering temperatures. A typical series of the values of the amplitude and phase of the first term of the characteristic equation is given in a graph plotted in polar coordinates using the value of the specimen drawn at 500 C. as the zero for the polar coordinates.

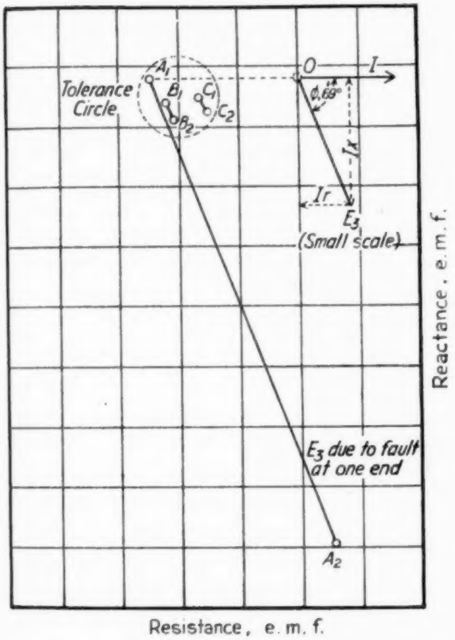


FIG. 9.—Electromagnetic Measurements on Tool Steel Rods Examined for Flaws from Center to Both Extreme Ends.

The graph in polar coordinates shows a flaw when it passes outside of the tolerance circle with the phase angle shown.

It should be noted that the sample nominally tempered at 620 C. does not occupy the expected position. The other tests showed that the samples tempered at 620 C. are proportionally located between the samples tempered at 600 C. and those tempered at 650 C. The obvious conclusion to be drawn is that this particular rod had been tempered at 602 C. instead of 620 C. due to some unexplained error.

If the rod had been cold worked by bending, for instance, the electromotive force value would not lie on the line with the other samples, which have a phase angle of −35 deg., since the phase angle of cold work is −165 deg. and this would cause the point to appear below the line of the other samples.

There is a divergence, also, in the case of the sample quenched but not tempered. This could not be investigated at the time. There seems to be a possible explanation on either metallurgical or physical grounds.

A progressive change in the tempering temperature is accompanied by a corresponding progressive change both in the electro-magnetic quantities and in the steel structure. This holds for a temperature range from 650 to 500 C. where the steel structure is so fine that the microscope can no longer be used. For tempering between 500 and 100 C., the structure is entirely outside of the limit of resolution of the microscope.[6]

It is generally believed that the structure of the steel also changes continuously after it becomes too fine for resolution by the microscope. Certainly there is a progressive change in the physical characteristics of the steel. The electro-magnetic measurements, made with an accuracy of 0.1 per cent, will probably give results which reflect any significant change which occurs in the structure of the steel.

Detection of Flaws in High-Speed Tool Rods:

As a typical instance of the inspection of material for cracks, seams, or other

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[6] At particular tempering temperatures, depending on the analysis and method of treatment, there is a very complex, but completely consistent, relation between the resistance and reactance components of the changing electromotive force. This is accompanied by corresponding changes in Rockwell hardness, Charpy impact test values and tensile strength. It is believed that the steel changes indicated are of great importance but in Fig. 8 such a region is not shown, probably because the temperature differences between points is too great to bring out the critical character of tempering effects occurring within certain narrow temperature ranges.

physical flaws, mention is made of the examination of cold-drawn rods. Frequently an inspection of a section cut from each end of every rod is required by the customer, since cracks sometimes develop during treatment and after regular inspection in the mill. This inspection is expensive in time and material without any assurance that there are no cracks in the rods between the inspected ends. The rods in question were cold-drawn, annealed, drastically cold worked, centerless ground to 0.594 in. in diameter. This material is subject to the formation of cracks during mill operations. The results of measurements made on three similar samples are plotted in polar coordinates in Fig. 9.

The tests of each rod are made at the extreme ends, the results from the two ends being designated in Fig. 9 by subscripts 1 and 2. The plotted values are joined with a straight line for convenience, although an unrecorded survey shows that the intervening points would make a continuous line but by no means a straight one. A circle drawn around the values given by the normal rods becomes the tolerance circle.

The values of the electromagnetic quantities lying outside follow a line which makes a phase angle of -69 deg. which is characteristic of a crack or seam in the material. As the rod passes through the coil the electromagnetic values leave the tolerance circle when the crack reaches the coil.

The tests at the extreme ends were made by using a balancing wave which neutralized the end effect. For such commercial work the correct balancing wave for the center of the tolerance circle can be recorded for each type of steel used. As they come through, a few at a time, for testing, the shift in the zero can be made promptly—taking no longer than tuning in a radio to a known broadcasting station.

CONDITIONS SURROUNDING THE TEST

1. The material should be of uniform analysis and uniform dimensions in the run of the material, for rapid testing in mill operations.

2. In any sample to be tested, there should be no abnormalities running uniformly throughout the run of the material, as for example a crack.

3. No two abnormalities should completely overlap throughout the run of the sample to be tested. This condition has not yet been encountered.

4. Disturbing effects should be evaluated if they are found to be of sufficient relative importance to be objectionable, for example, temperature differences of samples.

5. In laboratory tests it is assumed that all of the conditions are under control but in mill operations it is necessary to note the extent to which the conditions vary and to be sure that no significant error could be caused, for example, erratic tension.

6. Internal stresses must be considered but they have never been found to vary enough in the run of a sample to affect the results of the electromagnetic measurements of differences due to seams or other abnormalities in the material.

7. No physical standards of the material are needed but one or more unrelated test specimens should be kept on hand to use occasionally as a check on the condition of the apparatus.

CONCLUSIONS

The illustrations given cover only a small part of the work, but they are characteristic of the results that have been obtained. The fundamental assumption has been verified in every case examined throughout the whole range of laboratory investigations and commercial applications of the method.

Since commercial work must be standardized and carried on at high speed, without special adjustments, under the conditions found in operating mills, the variety of applications that can be made is limited but still leaves a wide field in which there can be profitable employment of this method of testing.

Research investigations have made successful use of the complete laboratory equipment in previously unexplored fields. The possibility of a further extension of the balancing wave method of testing seems assured.

Among applications to research which have not been attempted, involving problems of very considerable importance, the following are mentioned as they could be undertaken with confidence in view of comparable problems that have been completed with success, although not included in this paper:

1. Identification and measurement of the structural changes in steel by following the process continuously at the temperatures at which the changes are taking place.
2. Determination of the relation between the structure of steel rails and the formation of transverse fissures which later appear when the rails are subjected to the normal operating stresses.
3. The measurement of structural changes accompanying the progressive approach to fatigue failure.

The previous attempts to locate and measure such changes, before the lesions in the material begin to appear, have not been successful. The sensitivity and accuracy of the balancing wave method, however, offers a possibility that should be investigated.

DISCUSSION

Mr. Theodor Zuschlag.[1]—I should like to ask Mr. Kinsley a question with reference to the statement on page 37 of his paper: "In the method herein described, there is introduced into the circuit a balancing wave of electromotive force, composed of the controlled output of phase shifting transformers, which can completely neutralize any or all of the components of the complex electromotive force in the testing circuit. By balancing the effect of the disturbing factors, it is possible to make correct interpretation of the remaining electromotive force in terms of the structure and physical properties of the material." My personal experience, based upon the magnetic inspection of thousands of tons of steel, indicates that this statement is too optimistic. I have frequently encountered materials of entirely different chemical analysis which nevertheless produced exactly the same type of readings when tested magnetically. I have also seen materials which contained well-defined mechanical defects that did not show up to any extent in magnetic investigations. These cases, of course, are exceptions but, being facts, I believe they justify the statement that ambiguous results are not impossible in inspecting material by magnetic testing methods. From this viewpoint I wonder whether it pays to apply the tedious laboratory procedure of magnetic testing as proposed in Mr. Kinsley's paper?

Mr. E. E. Thum.[2]—I have just one remark to make of a practical rather than theoretical nature. I know that many attempts have been made to promote the use of electrical or magnetic tests for inspection processes. The greatest success I have seen has been in the manufacture of safety razor blades, where the electrical characteristics of the steel strip are used as a means of estimating the relative condition of the material at various points in the process of manufacturing, as compared with the characteristics of a short length taken as a standard and known to be of the required condition. In a continuous process of this sort very close control can be had, but in every case that I know of electromagnetic tests are used purely as a method of comparison against a standard. If the material under test balances against the standard sample, everything is fine; if there is a very great deviation, steps are immediately taken to find out to what the deviation is due—these steps involving the more conventional investigations by calipers, hardness testers, and microscope.

Mr. Carl Kinsley.[3]—Replying to Mr. Zuschlag, if the hysteresis loop is examined with the care employed in this method of testing its characteristic form is expressed by the derived electromotive force values which include the higher harmonics. The methods previously used give measurements which do not include the phase relations and do not consider quantitatively anything except the fundamental term of the electromotive force, due to the character of the instruments employed. The establishment of an exactly located zero and a complete balance of the electromotive force in the circuit is an essential part of this method of testing. When the measurements are made with these or equal precautions, I believe there will

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[1] Chief Electric Engineer, Magnetic Analysis Corp., Long Island City, N. Y.

[2] Editor, *Metal Progress*, American Society for Metals, Cleveland, Ohio.

[3] Consulting Engineer, New York City.

be found no lack of concordance with the general statement that "the hysteresis loop uniquely characterizes any ferromagnetic material with respect to its structure and physical condition."

When making commercial applications of this method of testing, it is possible to simplify the apparatus and the completeness of the tests applied without sacrificing the necessary accuracy. To prevent anomalous results the material must be uniform and the specific manufacturing processes carried out in accordance with definite specifications. Magnetic analysis tests show when such specifications have not been followed as well as the presence of flaws and other imperfections in the material. I have examined many thousand feet of material and have found no lack of agreement with the general statement.

The question raised by Mr. Thum in regard to the necessity for using a standard has been discussed in part in connection with Fig. 5. Referring to this figure it will be found that the location of the zero when air alone is in the coil is shown on the upper part of the sheet. All of the specimens are then located with reference to this fundamental zero and thus with reference to each other by the electromagnetic measurements. A secondary zero can be used at any convenient point as was done here by introducing into the circuit a balancing wave which reduced the electromotive force exactly to zero when specimen F_1 was present in the testing coil. If another specimen is then substituted for it, specimen G for instance, the electromotive force vector appearing in the circuit will characterize the difference between the two specimens. This is shown on the graph and marked G. The phase angle shown, −34 deg., is characteristic of the difference in structure due to annealing. Any other specimen with a different amount of annealing, but otherwise like G, will lie on a continuous line having this phase angle. Another sample, specimen F_2, was found to nearly coincide with F_1 but was slightly off from the annealing line and so indicated a physical difference. The presence of a little cold work alone would have shown a phase of +165 deg. (or its opposite −15 deg. instead of the actual −9 deg.). It is probable, therefore, that there is present also a slight difference in annealing. But the total difference would come within the allowable, or tolerance, variation so that the specimen was acceptable. (Computed graphically, the assigned values of the additional electromotive forces due to the cold work plus annealing differences equal the measured value,

$$-6.75/\underline{-15^\circ}+1.63/\underline{-34^\circ}=-4.3/\underline{-9^\circ}$$

as given by the first term of the complete equation No. 7.) Such an analysis is not possible except when the zero has been obtained so that the phase angle is known accurately.

In the commercial illustration given by Mr. Thum any deviation from the normal would cause a change in the electromotive force of the testing coil and so unbalance the testing circuit. Its amount and direction from the secondary zero of its vector would indicate the change which had occurred in the steel structure. The change in its physical constants would be immediately obtainable from the calibration curve of the material being used. Decarburization would be shown as a structure change and it could be identified by using one of the higher harmonics, the seventh for instance, and its depth by the instrument reading. A correction in the manufacturing process, such as its speed or temperature, could be made at once without obtaining the physical constants. In all commercial work it is necessary to have the apparatus initially adjusted by one familiar with its method of operation before turning it over to an untrained assistant for testing service.

A PROPOSED STANDARD CLASSIFICATION OF GRAPHITE IN GRAY CAST IRON

By W. E. Mahin[1] and J. W. Hamilton[2]

Synopsis

The need has been pointed out for a metallographic system of classification of the graphite phase in gray cast iron. Two proposed standard charts have been set forth. One of these in serving to identify graphite flake size is similar to the A.S.T.M. grain size chart for steels.[3] The other represents four types of association of graphite flakes differing from each other in their distribution or orientation. A technique for the application of these two charts has been proposed with emphasis laid on the use of suitable magnifications in order to produce a record representative of the entire area of the micro-section.

Introduction

Probably no single development of recent years has been of more importance to our fundamental knowledge of ferrous metallurgy than the work on austenite grain size in carbon and low-alloy steels. This work began about fifteen years ago with the realization that grain size was perhaps as important as the chemical analysis in determining the properties and reaction to heat treatment of steels. During the ensuing years, the work of many investigators in adopting a standard scale for the measurement of grain size; in studying the quantitative effect of grain size on hardenability, impact strength, and other properties; and in studying the laws of grain growth and in developing the means of control of grain size through melting practice has accomplished much toward establishing the metallurgy of steel as an exact science of untold value in its application to industry.

Through the latter part of this period of development in the metallurgy of steel, cast iron also has been a subject of much investigation. Studies of the effects of composition and of raw materials, melting methods and solidification rates have resulted in an ability to provide desirable combinations of mechanical properties for each of a wide variety of applications. The mechanism is, at least in part, control of graphite flake size. This valuable work has been handicapped, however, by the lack of a rational system for classification.

In the present work it is not desired to deal with either the control or the effect of graphite flake size. It is, however, desired to point out the variations in flake size, orientation, and distribution that may exist, and to propose the necessary standards and technique for their measurement.

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[1] Metallurgical Engineer, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.; formerly Research Metallurgist, Vanadium Corporation of America.

[2] Research Dept., Vanadium Corporation of America, Ann Arbor, Mich.

[3] Standard Grain Size Chart for Classification of Steels (A.S.T.M. Designation: E 19 – 33), 1936 Book of A.S.T.M. Standards, Part I, p. 761.

GRAPHITE FLAKE SIZE

Figure 1 shows a chart, in some respects similar to the A.S.T.M. grain-size chart for steels, composed of eight photomicrographs at a magnification of 100 diameters. The numbers 1 to 8 designate a series of progressively shorter flakes and, in the authors' experience, cover the range of flake size obtainable in sand-cast, gray-iron castings having a section diameter or thickness of from $\frac{1}{8}$ to 6 in. or larger. It is interesting to note that if the sizes of the A.S.T.M. grain-size chart for steels are transposed into a linear dimension, that is, mean grain diameter, the limits of the range obtained are about 1 in. and $\frac{1}{8}$ in., respectively, or in a ratio of 8 to 1. In the proposed flake-size chart, however, it has been found necessary to cover a range of from 4 in. to $\frac{1}{16}$ in. or in a ratio of 64 to 1. An important result is that the difference in size between adjacent numbers in the latter scale is appreciably greater.

It is interesting to observe at this point that an over-all range in size of austenite grains in steel comparable to the range in size included within the proposed flake-size chart actually is obtainable. Extreme refinement and exaggerated grain growth in steel may lead to austenite grains $\frac{1}{16}$ in. in diameter or smaller on the one hand and 4 in. in diameter or larger on the other.

In the course of preparing the present graphite flake-size chart, a considerable number of micro-sections was examined, representing a rather wide variety in as-cast section size, and in chemical analyses of irons. Mixed flake sizes, similar in a way to mixed grain sizes in steels, were found in many of these and it was deemed necessary to record all findings as proportions of a given size rather than merely attempting to show over-all size ranges within each specimen. This practice, commonly used also for steels of mixed grain sizes, seemed logical because of the probability of thus obtaining more exact correlation with the mechanical properties of the cast irons in question.

The estimation of proportions of predominant flake sizes would be extremely difficult if there were such a thing as random distribution of a wide range of flake sizes. In the authors' experience, however, mixed flake sizes usually exist as groups or areas composed largely of one size with the balance largely of another size. These areas have been found to fall into two general classes: (1) dendrites whose size may vary widely, and (2) rosettes or rounded groups $\frac{1}{64}$ in. or larger in actual diameter.

Since graphite flakes, like any other micro-constituent of metals, are three dimensional, naturally a certain amount of variation in flake size will be found in the most uniform cast irons. Each flake actually is more or less of a saucer-shaped particle and the particular section observed is purely a matter of chance. With an entirely random orientation of particles, this leads to no particular difficulties in arriving at an estimated mean size. However, in such a case as that illustrated in Fig. 2, type D, where the flakes are all oriented in restricted planes, it may become necessary to use a range of sizes for a single area.[4]

GRAPHITE TYPE CHART

Questions of distribution and orientation have been encountered in discussing the estimation of sizes and proportions of sizes. In order to include the two former factors in the classification, a chart of standard graphite types is proposed. In Fig. 2 are shown four types of graphite, varying from each other in

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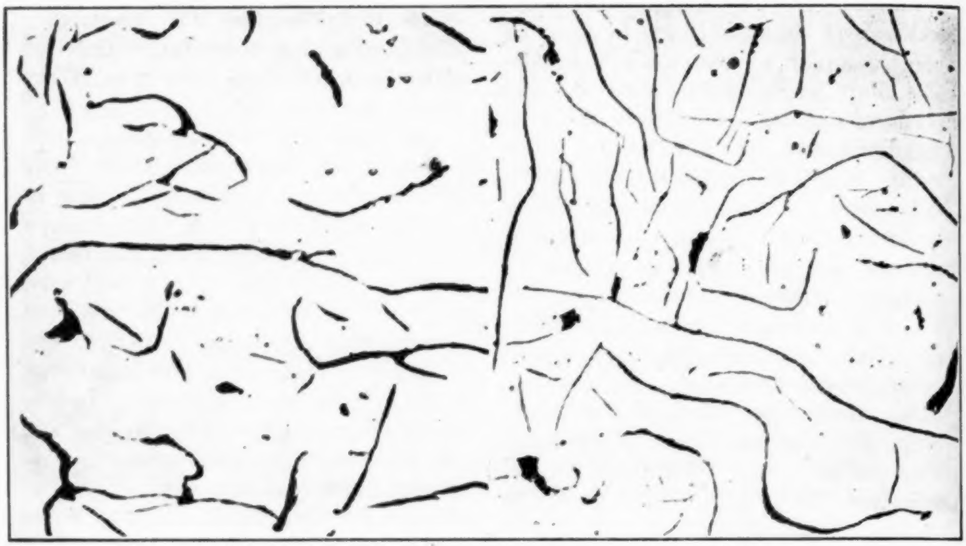
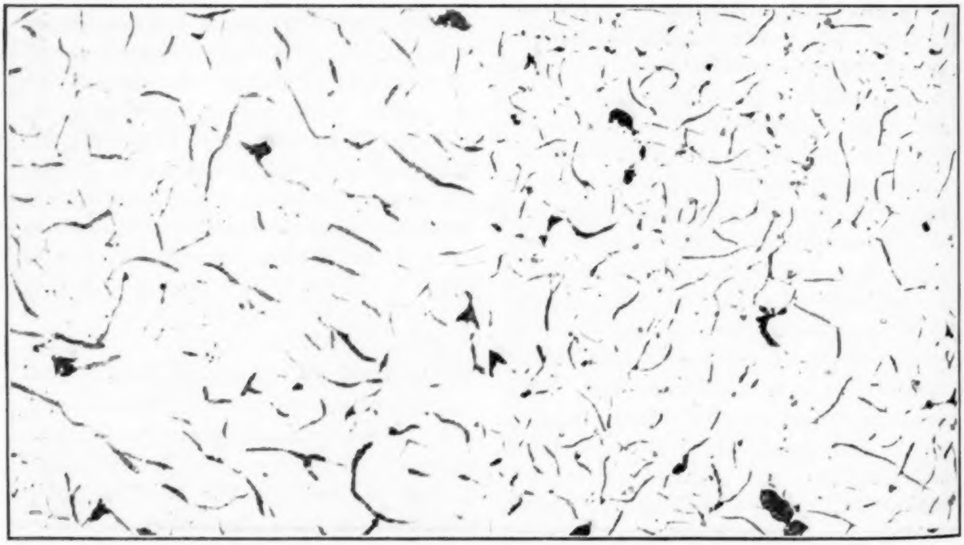
[4] It is believed, however, that the actual characteristic size of this zone is nearly that of the largest flakes seen because all other flakes probably consistently have been cut through a short axis.



No. 1
Flakes 4 in. or more in length.

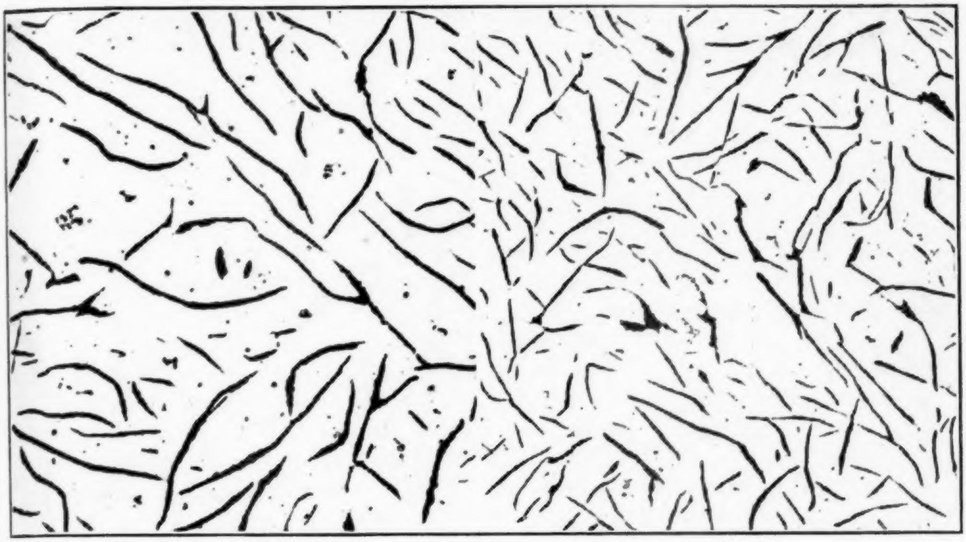
No. 2
Flakes 2 to 4 in. in length.



No. 5
Flakes ¼ to ½ in. in length.

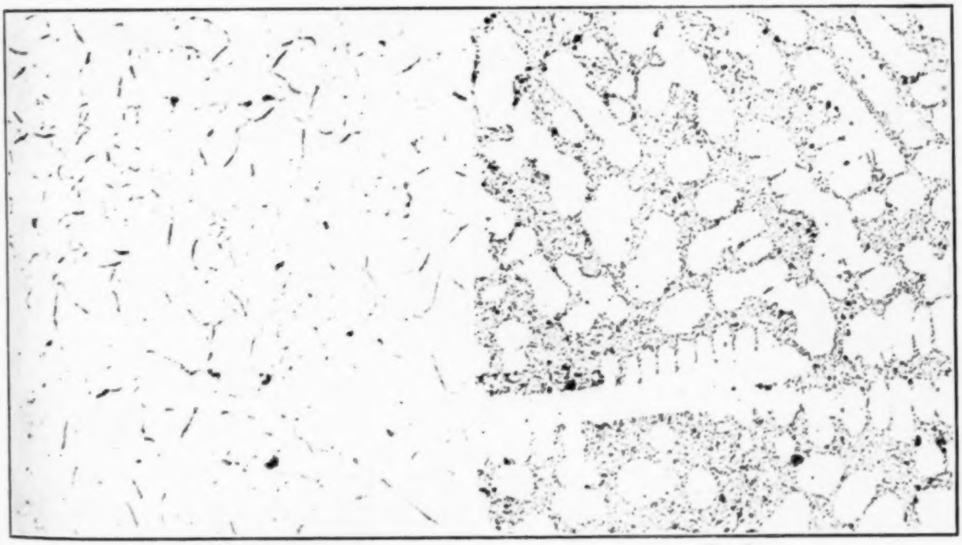
No. 6
Flakes ⅛ to ¼ in. in length.

FIG. 1.—Graphite



No. 3
Flakes 1 to 2 in. in length.

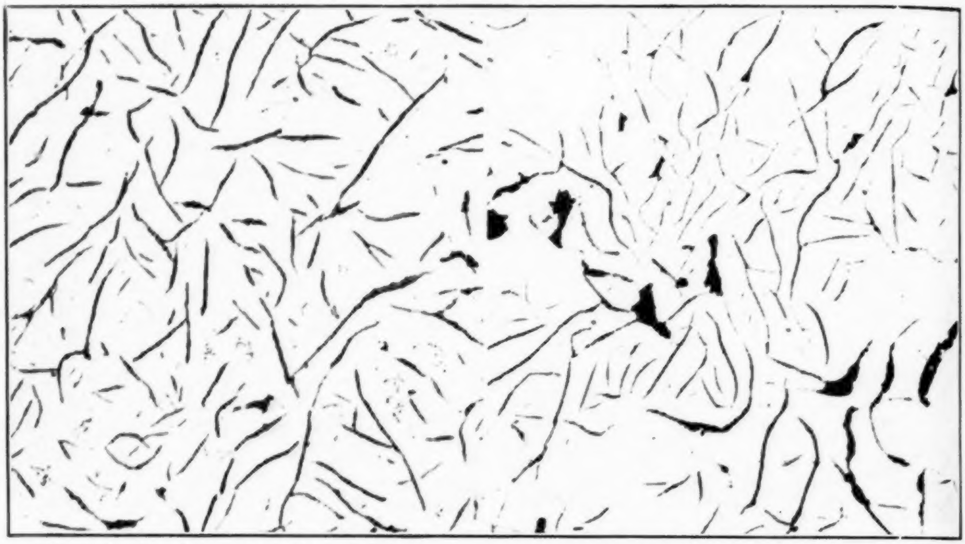
No. 4
Flakes ½ to 1 in. in length.



No. 7
Flakes $\frac{1}{16}$ to $\frac{1}{8}$ in. in length.

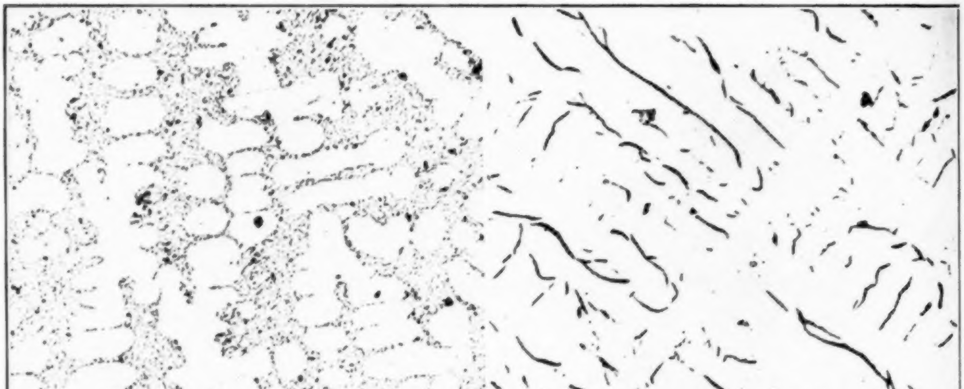
No. 8
Flakes $\frac{1}{16}$ in. or less in length.

Flake-Size Chart (× 100).



Type A—Random orientation of flakes uniformly distributed.

Type B—Random orientation of flakes grouped in rosettes.



Type C—Random orientation of flakes grouped in dendrites.

Type D—Dendritic orientation of flakes.

FIG. 2.—Graphite Flake-Type Chart (× 100).

the form of distribution and orientation of typical gray-iron graphite flakes. If it were desired to include the temper carbon type of graphite found in malleable irons, a fifth could, of course, be added and thus compose a system for classifying graphite in all cast irons.

SUITABLE MAGNIFICATIONS

Because of the tendency of graphite flakes to segregate into groups each of which may have a characteristic flake size, it is important to note that the direct use of the graphite flake-size chart at 100 diameters for comparison with projected images of specimens at the same magnification was rarely practiced in this work. To have done so would have made the estimation of proportions of different sizes highly difficult because of the relatively small area that could be examined at one time. To illustrate this point, Figs. 5 and 6 show at 100 diameters two areas which might represent greatly different cast irons. Actually, however, they were taken adjacent to each other from the same iron and both areas are shown in Fig. 4 at 25 diameters.

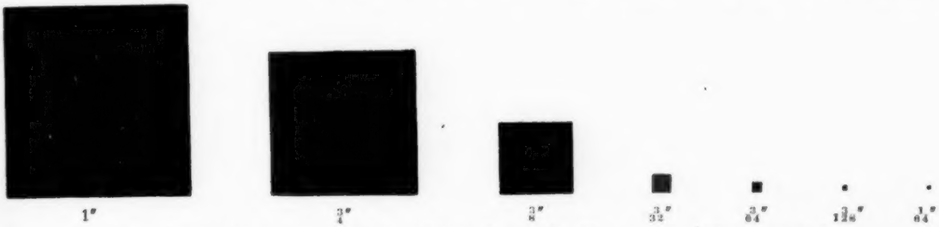


FIG. 3.—Comparison Standard for Observing Flake Size at Magnifications of 25.

TECHNIQUE OF EXAMINATION

The desirable procedure seemed to be the use of as low a magnification (and as wide a field) as could be used without too great a sacrifice in the accuracy of the measurement. In general, the accuracy of the measurement was aided by the rather wide difference between adjacent numbers on the scale.

Accordingly a method has been developed (found also applicable to mixed grain sizes in steels) involving the use of a wide-field binocular microscope at comparatively low magnifications. With a magnification of 10 to 25, all or much of the surface of an average microsection may be seen at one time. Flake-size readings are then obtained by comparison with a standard piece mounted beside the specimen on the stage of the microscope. This standard may be merely a small polished block of stainless steel near one edge of which a series of eight small holes or marks have been inscribed with diameter or length corresponding to the mean actual length of the eight flake sizes (see Fig. 3). With proper illumination, in this case obtained with the small incandescent bulb and reflector supplied with the microscope, comparisons between the standard and graphite flakes in the specimen are comparatively simply made, and the exact magnification used is unimportant.

Having scanned the specimen and noted the proportion of each size and type by the method just described, the flake-size chart and the type chart and a magnification of 100 diameters may be used if desired for checking the accuracy of the measurements and observations of type that have been made at lower magnification. This will be especially desirable for the extremely small sizes of graphite flakes.

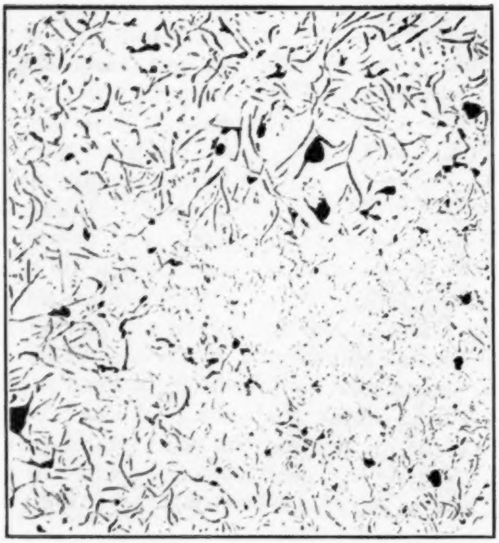


FIG. 4.—Variable Flake Size of the Dendritic Type (× 25).
60 per cent Size 4 Type A. 40 per cent Size 7 Type C.

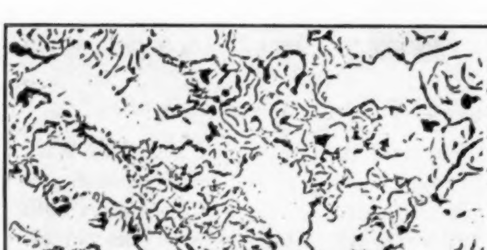


FIG. 5.—Small Graphite Flakes Shown in Fig. 4 (× 100).

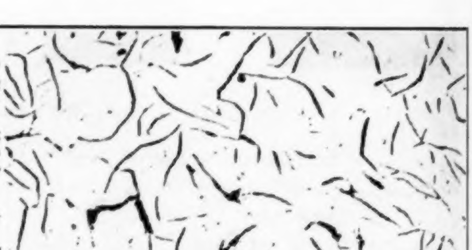


FIG. 6.—Large Graphite Flakes Shown in Fig. 4 (× 100).

One further consideration is that of segregation according to physical shape or location in the casting. A condition similar to the structural differences in case-hardened steels may exist, wherein a part of the iron will be distinctly different from other parts due to abrupt variations in cooling rate. In such cases two or more such structures in the same iron should be differentiated by classifying them separately as if for different specimens.

The standard scale used in this work was made in the following manner. On a duplicate piece of the stainless material used for the scale, an experiment was performed in order to correlate Vickers[5] load with impression size on this particular material. An ordinary set of gravimetric balance weights was used for obtaining various loads and the experimental results were plotted as weight against length of the side of the square impression. Once this was done, the desired lengths were interpolated from the curve, checked individually for accuracy, and finally the desired series of square impressions was imposed near the edge of the other block. In Fig. 3 is shown the appearance of the standard at a magnification of 25 diameters.

A special eyepiece similar to a grain-size ocular now available on the market for steels could, of course, be developed in conjunction with a standard graphite flake-size chart. Although such a device would require a fixed magnification for actual measurement of size, it could be calibrated at either 25 or 100 diameters and still be used for the estimation of proportions at any suitable magnification. Such an ocular probably would be most easily utilized if the scale were composed of a series of circles or squares of appropriate diameters or length of sides.

CONCLUSION

It is hoped that this brief presentation will stimulate discussion among those numerous metallurgists interested in the advancement of the metallurgy of gray cast iron, and furnish some impetus to the general adoption of a standardized method for studying and recording the graphite variable in this important engineering material.

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[5] A Vickers hardness testing machine was used.

DISCUSSION

Mr. J. F. Johnson[1] (*presented in written form*).—The authors have presented a classification of graphitic carbon in gray cast iron, which, when supplementing other factors, should be instrumental in securing iron with definite properties.

While the use of low magnification permits examination and classification of relatively large areas accurately, it is believed that for most types of work a micrograph of the unetched specimen at 100 diameters, taken as representatively as practicable, is sufficient. Micrographs of the etched specimen at both 100 and 1000 diameters are also desirable, however, to supplement the information gained from the graphite pattern.

To illustrate the use of micrographs at 100 diameters for direct comparison the photomicrographs shown in the accompanying Figs. 1 and 2 are presented. Both specimens are from cast iron snap locomotive piston valve rings of a similar mixture, but with different methods of casting. The valve bushing or liner in which the rings operate is also of similar mixture, but necessarily a cylindrical casting.

Locomotive Snap Piston Valve Rings, 14-in. Diameter, $\frac{5}{8}$-in. Section.

	Individual Ring Casting	Tube Casting, 18-in. Length
Brinell hardness....	218	182
Fracture..........	Mottled	Gray
Mileage in service...	10 000 approx.	132 663

The rings are subjected to temperatures of superheated steam ranging from 600 to 715 F., where fluid film lubrication is sometimes replaced by boundary conditions. When lubrication is virtually displaced, experience has shown that a cast iron ring with a fully lamellar pearlitic structure, and moderate graphite flake size, has better wear resistance than where considerable free ferrite is present. The presence of cementite results in early scoring of both the ring and the liner.

The micrographs of Fig. 1 show a dense structure, small graphite flake size similar to No. 7 of the authors' classification, with considerable cementite. The fracture was highly mottled.

The iron of Fig. 2 has a more open structure with a moderate graphite flake size equivalent to No. 4 of the proposed classification. The valve rings of this material had sufficient tension to provide good wall pressure and the wear resistance was excellent.

The Chairman of Subcommittee VII, of Committee A-3, on Microstructure of Cast Iron pointed out some years ago the need for a system of correlating microstructure with other properties. The present paper adequately classifies the graphite phase.

The photomicrographs were available through the courtesy of E. J. Edwards, Chief Metallurgical Engineer, American Locomotive Co.

Mr. G. F. Comstock[2] (*presented in written form*).—The paper by Messrs. Mahin and Hamilton is timely and should result in a useful standard for comparing cast iron structures.

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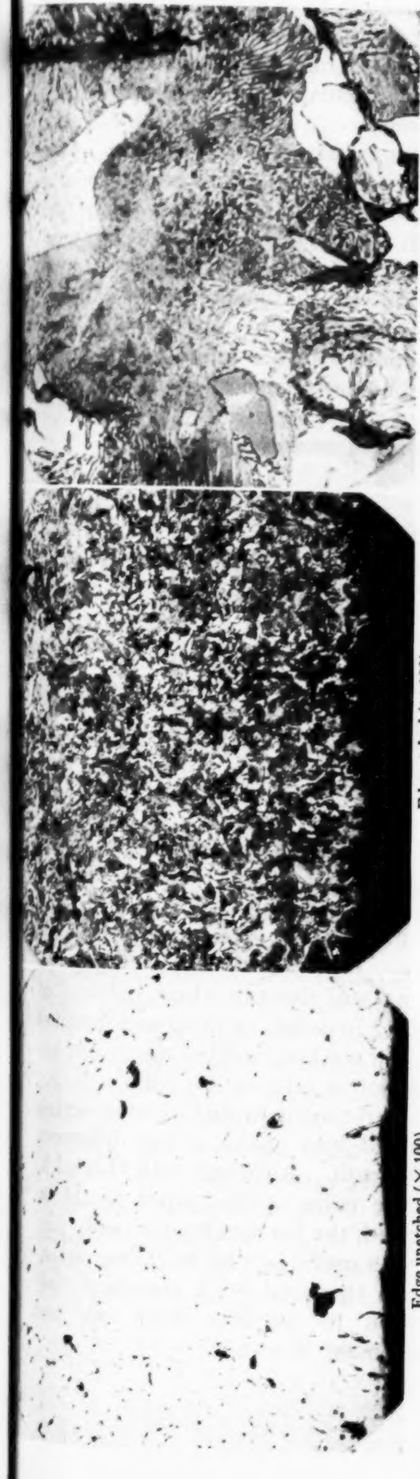
[1] Inspector of Tests, Florida East Coast Railway, St. Augustine, Fla.

[2] Metallurgist, The Titanium Alloy Manufacturing Co., Niagara Falls, N. Y.

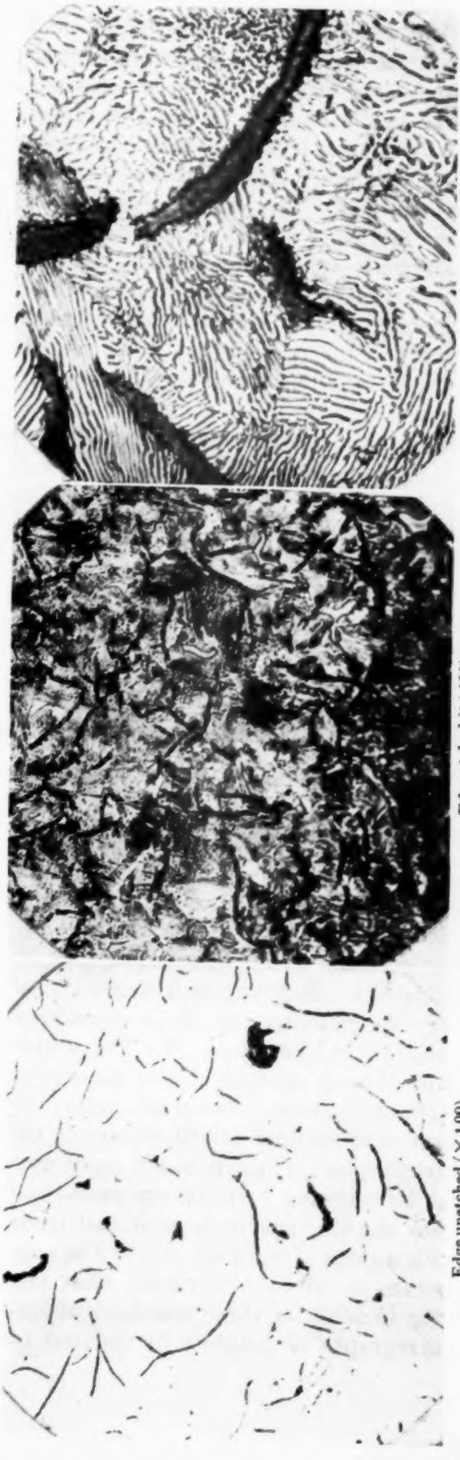


Adjacent to edge (× 1000).

Edge etched (× 100).

Edge unetched (× 100).

FIG. 1.—Cast-Iron Piston Valve Packing Ring, Individual Casting. Etched in 2 per cent Nital.



Adjacent to edge (× 1000).

Edge etched (× 100).

Edge unetched (× 100).

FIG. 2.—Cast-Iron Piston Valve Packing Ring, Tube Casting. Etched in 2 per cent Nital.

The titles assigned to the various illustrations in Fig. 1, however, do not seem to be correct. Photomicrograph No. 1, for instance, shows not a single flake as long as 4 in., yet the title states that the flakes in it are at least that long. Similarly, No. 2 shows flakes up to 2 in. in length, No. 3 up to 1¼ in., No. 6 up to ⅜ in., and No. 7 up to ¼ in. There seems to be quite a gap between No. 7 and No. 8, but probably that is permissible. Before Fig. 1 is issued as a workable standard, the titles should be corrected to agree with what is actually shown.

A good part of the paper is concerned with the matter of reconciling the chosen magnification of 100 diameters with practical applications which necessitate a lower magnification, such as 25 diameters. But why is it necessary to use 100 diameters at all in examining graphite in cast iron? To the writer this seems a mistake which, strangely, is too often made. In our laboratory we have standardized on 50 diameters for this purpose, which is much more useful for showing a really representative view of the graphite in most cast irons with a print of ordinary size. The suggestion is offered, therefore, that the magnification of these standard photomicrographs of graphite be reduced to 50 diameters to make them more practically useful.

Another point which needs careful discussion and possibly standardization is the method of polishing the specimens for comparison of the graphite. The proper polishing of cast iron is more difficult than that of steel, and even though it was not necessary to discuss this point in connection with the standard grain-size photomicrographs of steel, it would be very advisable to do so in connection with these photomicrographs of graphite. The method of polishing is really of fundamental importance, for the graphite may be made to appear either coarser or finer, according to the way the specimen is prepared for examination. It is to be hoped that the authors will describe their method of polishing in detail, or propose a detailed standard method, and too much emphasis cannot be laid on the details since a slight variation in procedure or materials employed may make a big difference in the result. Although with this addition the value of the paper would be enhanced, the authors have already performed a useful service in calling attention to the need for a standard classification of graphite flake size and arrangement.

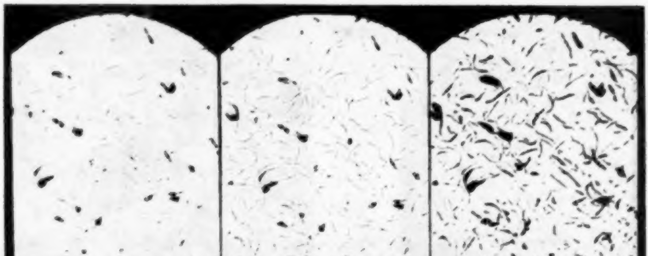


FIG. 3.—Identical Field in Gray Cast Iron Polished by Three Slightly Different Methods.

MR. C. K. DONOHO[3] (*presented in written form*).—The effect of method of polishing upon the appearance of graphite under the microscope is an important consideration in discussing graphite size. The accompanying Fig. 3 shows three views of the identical field in a sample of gray iron when polished by slightly different methods. The left side of the figure shows the sample as finished on a felt-covered polishing wheel using as the polishing compound powdered alumina suspended in stearic acid. The wheel was almost dry, as just enough water was used to prevent sticking. The improved definition of the graphite in the middle of the figure was produced by just touching the sample as previously polished to a polishing wheel covered with No. 00 carborundum paper to remove so-called amorphous iron smeared over the surface. To avoid scratching, the carborundum paper used for this purpose was prepared by rubbing with an old sample to break down the larger protruding grains. The "dug-out" appearance of the graphite on the right side of the figure was produced by wet polishing on a felt wheel with alumina powder suspended in water. The original magnification was in each case 100 diameters on a 6-in. diameter field.

The distinctly different appearances of these three micrographs of the same spot on the sample show the need for some system of definite requirements in the polishing of cast iron specimens before any suitable classification of the graphite structure can be universally applied. In numerous examples in the literature showing micrographs of "fine" graphite contrasted with "coarse" graphite, the only real difference may often be in the preparation of the specimens photographed. A micrograph such as that on the left-hand side of the figure would seem to indicate a rather high-strength cast iron while a micrograph such as that on the right-hand side of the figure would appear to be that of a weak, coarsely graphitic iron, whereas, of course, both are from the same sample.

I should like to ask the authors what method of polishing they used to insure that the graphite was neither "dug out" nor "smeared over" and if they have any suggestions for a polishing method which will produce concordant results.

The micrographs shown were prepared by Mr. E. M. Whelchel of the American Cast Iron Pipe Co.

MR. BRADLEY STOUGHTON.[4]—I want to support very strongly what Mr. Donoho and Mr. Comstock have said about the necessity for some standard method of polishing, without which, of course, we cannot really get any true basis of evaluating the iron.

I think all of us who try to explain the high strength of a 60,000-lb. or 50,000-lb. cast iron on the basis of structure have failed to find a true correlation, and some method of getting a standardized description of structure may perhaps bring this into a more systematic basis, such as we have, for example, in the alloy steels. It seems to me, that if we could have a round-table talk on this subject in which a number who have been working on it could exchange data, we might really get a long step forward. I think it is a splendid thing that Messrs. Mahin and Hamilton have started by emphasizing these points.

MR. A. J. HERZIG.[5]—I am not quite certain that we are justified in paying so

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[3] Assistant Metallurgist, American Cast Iron Pipe Co., Birmingham, Ala.

[4] Director of Metallurgical Engineering, and Dean, College of Engineering, Lehigh University, Bethlehem, Pa.

[5] Chief Metallurgist, Climax Molybdenum Company of Michigan, Inc., Detroit, Mich.

little attention to the different forms of graphite. I see no great objection to classifying graphite as to size, but we must bear in mind that we have five or six different types of graphite formation and we must recognize that until there is some agreement as to the temperature and mechanism of formation of the various types, a graphite size classification would not only be of limited value but would most probably lead to erroneous over-emphasis of the graphite size factor. The early discussion of the ambiguous correlation of grain size and abnormality in steel is a situation which we should attempt to avoid in devising classification schemes for cast iron.

MESSRS. W. E. MAHIN[6] AND J. W. HAMILTON[7] (*authors' closure*).—Mr. Johnson has shown an interesting illustration of the effect of the microstructure of cast iron upon service life in a particular application. While the authors agree with him regarding the importance of the size and quantity of microstructural constituents other than graphite, it is believed that Mr. Johnson may have failed to appreciate the significance of the magnification used for classifying the graphite. The method described in the paper was designed to provide representative records of the microstructure of graphite without actually taking any photomicrographs. This, of course, is analogous to the method commonly used for evaluation of grain size in steels where the work of taking photomicrographs is eliminated simply by making reference to grain size numbers. The authors strongly recommend, however, the use of quite low magnifications for scanning the specimen, since it has been their experience that many cast irons are not sufficiently uniform to enable obtaining either a single representative photomicrograph or a flake size number. This lack of uniformity may easily be overlooked or at least incorrectly observed at 100 diameters or higher magnification. It is believed that this point has been amply demonstrated in the paper.

Mr. Comstock has questioned the necessity for using a magnification of 100 diameters for the proposed standard chart. This was done partly to adhere to the standard already established in the A.S.T.M. for grain size in steels and partly to gain optical resolution for the smaller sizes of flakes. It was pointed out in the paper that a magnification as low as 25 was not sufficient for studying the size of very small flakes of graphite. The authors doubt whether even 50 diameters would be sufficient for the latter purpose besides being somewhat excessive for enabling the scanning of variations within the specimen.

Mr. Comstock also has questioned the suitability of some of the photomicrographs used in the chart, and the authors agree with him that perfect examples of the range of flake length indicated have not always been obtained. However, a considerable number of specimens were examined and the portions photographed were selected with a great deal of care. The example used for size No. 1 could not possibly show a flake length of 4 in. when the maximum dimension of the photomicrograph as herein reproduced is only 2¾ in. The original photomicrograph did, however, show one flake 4 in. in length.

In order to improve on the present chart, it therefore would first be necessary to enlarge the over-all size of photomicrographs. Also, it might be desirable to replace some of the present pho-

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[6] Metallurgical Engineer, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.; formerly Research Metallurgist, Vanadium Corporation of America.

[7] Research Dept., Vanadium Corporation of America, Ann Arbor, Mich.

tomicrographs. The authors wish to emphasize, however, that this would require either a considerable amount of work by one or two individuals or else the cooperation of a number of laboratories interested in the problem. Again, it should be stated that the photomicrographs actually only are illustrations of a specific range in a linear dimension which is most accurately studied by actual measurement with some sort of an ocular, rather than by comparison with another photomicrograph.

Messrs. Comstock, Donoho, and Stoughton all have discussed the question of the importance of polishing. The authors would like to point out that Mr. Donoho's photomicrographs bear out rather well the authors' contention that flake length is not actually influenced by polishing technique. It is true that Mr. Donoho's photomicrographs present a different appearance. Careful observation, however, reveals that the difference is in apparent breadth rather than in length of the flakes. Although interest has been centered chiefly upon the methods of observing flake size, rather than its effect, it is not believed that the properties of cast iron would be affected by flake breadth nearly as much as by flake length.

It has been the authors' experience that various methods of polishing may be used to good advantage by various persons, but that as much depends upon the ability of the polisher as upon the particular materials and steps utilized in the polishing process.

The authors agree with Mr. Herzig that both the causes and the effects of various graphite flake formations and sizes require much study. They differ strongly with him, however, in his questioning the importance of standardized methods of measurement and observation of types until the cause and effects of these factors are more thoroughly understood. It is the authors' opinion that the latter work can proceed effectively only when suitable standards are generally available.

RELATION OF PROPERTIES OF CAST IRON TO THICKNESS OF CASTINGS

By H. L. Campbell[1]

Synopsis

Data are presented on the tensile and compressive strengths of irons cast in round bars of different diameter. The results of other investigations on this subject are reviewed. Experimental test data are summarized on a chart which indicates the relation of the tensile strength to the thickness of the metal in separately cast round bars. This characteristic of gray cast iron has special significance in the development of designs for iron castings.

The physical properties of the metal in gray iron castings are affected to a greater extent by differences in section than those of other cast metals. In heavy sections, which cool slowly in the mold, the strengths are less than in light sections which cool more rapidly. This situation is caused by differences in the proportion and condition of the graphite in the metal. Slow cooling promotes the separation of graphite and results in a decrease in the tensile strength of the metal.

An investigation has been made to determine the extent of the variations in the physical properties of different classes of cast iron with changes in the sections as cast. The plan for this investigation was to supplement the results of previous studies so as to cover all sections of cast iron from ¼ in. to 4 in. in thickness.

The results of a large number of tests of cast iron of different compositions in sections of 0.875, 1.20, and 2.00 in. are given in the 1934 Report of the Society's Committee A-3 on Cast Iron.[2] Boegehold made tests of specimens cast separately in rectangular sections from ⅛ to 1¼ in. in thickness after machining two sides of the cast bars.[3]

Healey obtained data on the tensile strength of the metal in a rectangular hollow casting having wall thicknesses of ½, 1, 2, and 4 in.[4] which showed that an average increase in strength of the metal in the ½-in. section was 11 per cent over that in the 4-in. section. Apparently, the heat in the heavier sections retarded the cooling of the metal in the lighter sections.

Mochel determined the tensile strengths of the metal in a cylindrical casting having sections of ½, ¾, 1, 1½, 2, 3, and 4 in. in thickness.[5] The tensile strengths decreased rapidly in the sections from ½ to 1 in. in thickness and

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[1] Metallurgical Engineer, American Hoist and Derrick Co., St. Paul, Minn.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 145 (1934).

[3] A. L. Boegehold, "Effect of Section Size on Physical Properties of Cast Iron," *Proceedings*, Am. Soc. Testing Mats., Vol. 30, p. 199 (1930).

[4] M. V. Healy, "Comparison of the Physical Properties of Different Sections of Cast Iron and of the Standard Arbitration Test Bar," *Proceedings*, Am. Soc. Testing Mats., Vol. 30, p. 206 (1930).

[5] N. L. Mochel, Discussion of Symposium on Cast Iron, *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part II, p. 145 (1929).

more slowly in changing from the 1-in. to the 4-in. section. The average increase in strength of the metal in the ½-in. section was 42 per cent over that in the 4-in. section.

Bornstein has reported the results of tension tests of cast iron of five different compositions in separately cast test bars having diameters of 0.75, 1.2, 2.0 and 3.0 in.[6] Gilligan and Curran in tests on iron castings up to 10½ in. in thickness found that the tensile strengths decreased very slowly when the sections of metal are larger than 4 in. in thickness.[7] MacKenzie's data on separately cast bars of different irons in sizes of 0.875 to 3 in. in diameter[8] show a considerable drop in the tensile strength of each iron as the section increases.

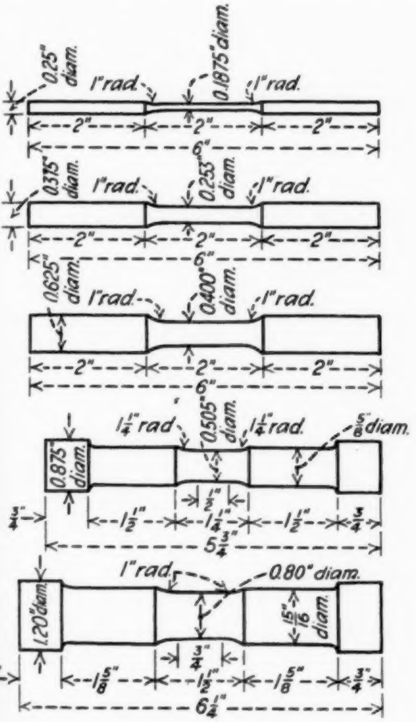

FIG. 1.—Tension Test Specimens.

Rother and Mazurie found that the tensile strength decreased as much as 53 per cent when the cast section was increased from 1 to 3 in.[9] Bolton's data show a considerable falling off in strength as the size of the cast bars increases.[10] MacPherran likewise reported a falling off in tensile strength with increase in sections of three types of iron.[11]

TABLE I.—CHEMICAL COMPOSITIONS OF HEATS OF CAST IRON.

Heat	Total Carbon, per cent	Silicon, per cent	Manganese, per cent	Sulfur, per cent	Phosphorus, per cent
No. 1	3.52	2.47	0.58	0.06	0.41
No. 2	3.50	2.58	0.59	0.07	0.44
No. 3	3.53	2.71	0.60	0.06	0.36
No. 4	3.26	2.62	0.64	0.06	0.32
No. 5	3.55	2.74	0.66	0.07	0.40
No. 6	3.43	2.61	0.60	0.07	0.38
No. 7	3.45	1.93	0.44	0.14	0.61
No. 8	3.55	2.06	0.52	0.13	0.47
No. 9[a]	3.30	2.46	0.90	0.10	0.18

[a] This heat also contained 0.50 per cent copper and 0.30 per cent chromium.

TABLE II.—AVERAGE TENSILE STRENGTHS OF SEPARATELY CAST BARS.

Diameter of Bars as Cast	Tensile Strength, lb. per sq. in.				
	0.25 in.	0.375 in.	0.625 in.	0.865 in.	1.20 in.
HEAT:					
No. 1	50 800	38 100	32 200	26 000	23 300
No. 2	43 800	34 800	34 900	29 250	23 750
No. 3	....	41 250	33 200	27 300	23 100
No. 4	46 300	36 150	32 100	26 000	21 700
No. 5	50 950	33 150	28 800	26 100	21 600
No. 6	44 600	....	35 200	31 600	27 200
No. 7	62 750	43 000	44 000	39 200	35 400
No. 8	57 000	44 000	42 600	36 200	32 100
No. 9	....	53 700	44 400	39 000	34 700

To supplement the results of tests

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[6] Hyman Bornstein, "A Comparison of Various Sizes of Test Bars Representing Cast Iron from Five Foundries," *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part I, p. 152 (1931).

[7] F. P. Gilligan and J. J. Curran, "Effect of Section on Tensile Strength of Gray Iron," *The Iron Age*, May 19, 1932, pp. 1106–1107.

[8] J. T. MacKenzie, American Cast Iron Pipe Co., private communication September, 1935.

[9] W. H. Rother and V. Mazurie, "The Strength of Cast Iron in Relation to Its Thickness," *Transactions*, Am. Foundrymen's Assn., Vol. 34, pp. 746–765 (1926).

[10] J. W. Bolton, "On Research Problems of the Gray Iron Foundry," *Transactions*, Am. Foundrymen's Assn., Vol. 36, pp. 469–512 (1928).

[11] R. S. MacPherran, "Effect of Section and Various Compositions on Physical Properties of Cast Iron," *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part II, p. 76 (1929).

reported previously by Committee A-3 on separately cast bars,[2] the present series of cylindrical test bars, 15 in. in length, having diameters of 0.25, 0.375, 0.625, 0.875, and 1.20 in. were cast of irons of different compositions. The dimensions of the tension test specimens prepared from the cylindrical bars are shown in Fig. 1. The molds for this series of test bars were prepared from an oil-sand mixture rammed in flasks made of 10-in. pipe. A pouring basin formed in a dry-sand core was placed on each mold which was poured with the test bars in a vertical position. The compositions of the nine heats used are given in Table I.

Special care was required in turning and testing the small cast-iron specimens. All specimens were turned from the centers of the cast bars and were tested in grips which insured central alignment of the loads. The average tensile strengths are given in Table II. A few specimens contained defects which necessitated the exclusion of the test results from the series.

The average results of the tension tests of the separately cast bars in sizes from 0.25 to 1.20 in. in diameter from the present series of tests, as given in Table II, and on larger sizes as reported previously are plotted in Fig. 2. The three standard sizes of test bars recommended in the A.S.T.M. Standard Specifications for Gray-Iron Castings

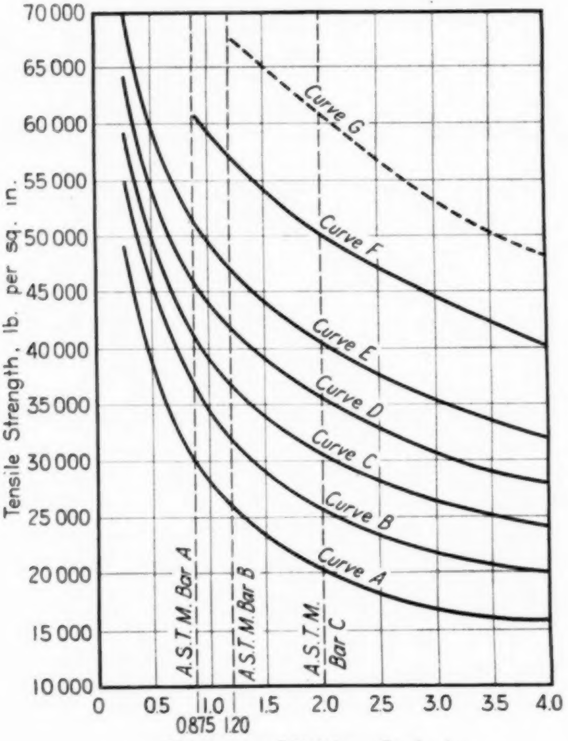

FIG. 2.—Tensile Strengths of Separately Cast Round Bars of Cast Iron

(A 48 – 36)[12] are shown. Each curve represents a different composition of cast iron. The curves are the approximate boundaries of the A.S.T.M. class C irons specified in A.S.T.M. Specifications A 48. For example, the area between curves *A* and *B* prescribes the range of tensile strengths of all class 20-C irons, which class includes those irons having tensile strengths between 20,000 and 25,000 lb. per sq. in. when cast in round bars 2.00 in. in diameter and tested in specimens 1.25 in. in diameter.

The variations in the tensile strengths with thickness indicated by any curve in Fig. 2 may be considered as the maximum to be expected. With greater mass of metal in a casting, even though the thickness remains the same, the cooling rate will be decreased and the tensile strengths will be less. In iron castings having sections of different sizes, the strength of the metal in any section will approach the strength of the metal in the next larger section to which it is connected, due to the heat in the heavier sections retarding the cooling of the metal in the lighter sections. Therefore, the slope of the curve showing the relation of tensile strength to thickness of an iron will be less when the cooling of any section is retarded by a greater mass of metal or by the heat from adjoining sections.

Relation of Compressive Strength to Thickness of Iron Castings:

Specimens machined from round separately cast test bars were tested in compression to determine the relation of compressive strength to the thickness. All surfaces of the specimens were carefully ground, with a length of specimen three times the diameter. The specimens were tested between hardened steel blocks, the upper block having a spherical seat.

The results obtained from the tests (Table III) indicate that the compressive strength increases as the thickness of the cast section decreases at about the same rate as the increase in tensile strength for the same cast iron.

TABLE III.—AVERAGE COMPRESSIVE STRENGTHS OF SEPARATELY CAST BARS.

Diameter of Bars as Cast.....	Compressive Strength, lb. per sq. in.				
	0.25 in.	0.375 in.	0.625 in.	0.875 in.	1.20 in.
HEAT:					
No. 1 ..	152 000	129 000	130 000	96 200	82 800
No. 2....	176 000	149 000	125 000	99 000	98 000
No. 5....	190 000	156 000	117 000	98 000	80 000

CONCLUSIONS

The tensile and compressive strengths of gray cast iron are dependent upon the size of the castings from which the test specimens are prepared. The metal in a light section has a higher strength than metal of the same composition in a heavy section. In giving the tensile strength of cast iron, it is necessary always to state the size of the casting from which the test specimen was obtained.

Acknowledgment.—The author gratefully acknowledges the assistance obtained from faculty research funds at the University of Michigan during the collection of the test data reported in this paper.

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[12] 1936 Book of A.S.T.M. Standards, Part I, p. 457.

DISCUSSION

Mr. J. T. MacKenzie.[1]—On behalf of Subcommittee XI on Methods of Testing, of the Society's Committee A-3 on Cast Iron, I should like to request Mr. Campbell to give us the details of the tests on the small specimens, which would be very interesting from the statistical standpoint. He notes, for instance, on page 68, that a few specimens contained defects and I think the committee would be very much interested in the actual number of defects in the various size specimens.

Mr. V. A. Crosby.[2]—I note with particular interest the author's work on compression testing, since most papers on this subject lack such data.

While the conclusions drawn by the author in the light of the investigations conducted are entirely justified, they would not, of course, apply to all compositions of gray irons. Frequent exceptions have come to our attention where high test irons were under consideration which showed higher tensile values in the larger sections than in the smaller sections; as shown by values given for bars A and B. A. S. T. M. machined tension bars, 0.800 in. in diameter were used in each case. An example is shown below:

Bar	Size, in.	Brinell Hardness Number	Tensile Strength, lb. per sq. in.
A	1.2	311	48 400
B	1.5	286	54 120
C	2.0	255	47 100

(All bars poured from same sprue.)

Mr. J. G. Pearce[3] (*by letter*).—Attention is called to additional data on strength-size relation of cast iron in various papers by the writer.[4] These mainly refer to the transverse test, which offers the advantage of testing bars on virtually any section that can be conveniently cast, with or without skin-machining. The establishment of size-strength curves in Britain met with the comment that bars were tested above and below the size limits for which the iron concerned would be used in practice, but where this is true the resulting curves are of great interest and value, and many castings exhibit wide variations in section. The effect of adjacent varying sections in the casting on strength is being explored.

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[1] Metallurgist, American Cast Iron Pipe Co., Birmingham, Ala.

[2] Metallurgical Engineer, Climax Molybdenum Co., Detroit, Mich.

[3] Director, British Cast Iron Research Assn., Birmingham, England.

[4] "Use and Interpretation of the Transverse Test for Cast Iron," *Journal*, Iron and Steel Inst. (British), Vol. II, p. 73 (1928); "Influence of Size of Section on the Strength of Gray Cast Iron," *Proceedings*, Inst. British Foundrymen (1928–1929); "Design of Engineering Castings in Relation to Tests," *Foundry Trade Journal*, January 24, 1929; "Correlation of Mechanical Tests for Cast Iron," *Transactions*, Am. Foundrymen's Assn. (1930).

A STUDY OF THE EFFECT OF SPAN ON THE TRANSVERSE TEST RESULTS FOR CAST IRON

BY J. T. MACKENZIE[1] AND C. K. DONOHO[1]

SYNOPSIS

This paper investigates the variation in results of transverse tests of cast iron caused by varying spans. Tests by previous investigators are discussed, as well as methods and results of new tests on rectangular bars of two different types of gray iron. By mathematical analysis of results, quantitative measure of the effect of changing the span to depth ratio is developed for bars of both round and rectangular section.

The conventional beam formulas for modulus of rupture and modulus of elasticity (so called) are based on certain mathematical assumptions known to be incorrect for a material such as cast iron. For round bars the conventional beam formulas may be simplified to:

$$\text{Modulus of rupture} = \frac{8WL}{\pi d^3} \quad \ldots (1)$$

$$\text{Modulus of elasticity} = \frac{\text{Modulus of rupture } L^2}{6\,d\,\text{def.}} \quad \ldots\ldots (2)$$

where W = breaking load,
L = length of span,
d = diameter of bar, and
def. = ultimate deflection.

For rectangular bars the equations are:

$$\text{Modulus of rupture} = \frac{1.5\,WL}{bd^2} \quad . (3)$$

$$\text{Modulus of elasticity} = \frac{\text{Modulus of rupture } L^2}{6\,d\,\text{def.}} \quad \ldots\ldots (4)$$

where b = breadth of specimen, and
d = depth of specimen.

Investigation of available test results, where cast iron bars were tested on varying lengths of span, brings out the fact that modulus of rupture and modulus of elasticity as calculated are not constant with varying span. Lacking information on rectangular bars, experimental work was carried out on strips from twc distinctly different types of cast iron which indicated the same general trends as brought out by the data on round bars in the existing literature.

ROUND BARS

A survey of the literature revealed a few series of tests on round cast iron bars which were of sufficient scope and accuracy to shed some light upon the subject under investigation. The type of equation used to express the variation of test results with varying span was that developed by Heller and Jungbluth[2] for deflection changes with span.

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[1] Metallurgist, and Assistant Metallurgist, respectively, American Cast Iron Pipe Co., Birmingham, Ala.

[2] P. A. Heller and H. Jungbluth, "Die Wandstärken empfindlighkeit getrennt gegossener Gusseisenproben und ihre Beziehung zur chemischen Zusammensetzung," *Archiv für das Eisenhüttenwesen*, Vol. 5, p. 75 (1934).

Modulus of Rupture:

Heller and Jungbluth[2] give some results on transverse tests of bars of 10, 20, 30, and 40 mm. in diameter. Bars of each size were tested on three different spans so that the ratios of span to diameter were, for each size bar, 10, 20, and 30. "Bending strength" per unit section area (Biegefestigkeit—comparable to modulus of rupture) were calculated, and expressed for each size bar (Table 5) in per cent of the value for ratio of span to diameter equal to 20. By this method of expressing results, the values obtained for the different diameter bars are comparable, since each value is a percentage of a value obtained from a bar of the same diameter.

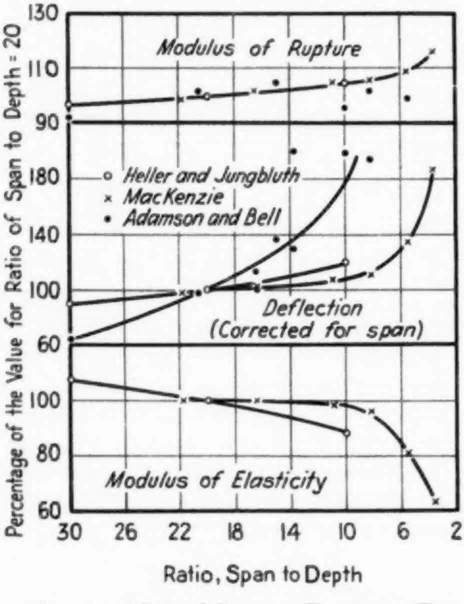

Fig. 1.—Effect of Span on Transverse Test Results of Round Bars.

The average values for all sizes and all irons tested for the three different ratios of span to depth are plotted in Fig. 1.

For comparison, the results of MacKenzie[3] recalculated as a percentage for values of span to depth equal to 20, are also plotted. In the same figure are shown results calculated from tests by Adamson and Bell.[4]

The deviation of these points from the 100-per-cent line represents the deviation of actual test values from the theoretical values. That is, by Eq. 1, modulus of rupture should be the same for a given iron, of given diameter, on any length span.

The equation of the line formed by the points calculated from the data of Heller and Jungbluth,[2] and MacKenzie[3] is found to be

$$\text{Percentage value} = \left(\frac{r}{20}\right)^{-0.07} \times 100$$

where r = ratio of span to depth

or

$$\frac{\text{Modulus of rupture}_{(r=x)}}{\text{Modulus of rupture}_{(r=20)}} = \frac{\left(\frac{x}{20}\right)^{0.93}}{\left(\frac{x}{20}\right)}$$

Therefore these data indicate that, in order to make the modulus of rupture independent of span, the term representing length of span indicated as being raised to the first power in Eq. 1 should be indicated as being raised to the 0.93 power.

Deflection:

Heller and Jungbluth[2] from very complete test data on deflection of bars for varying ratios of span to depth found that for values of r = 10, 20, and 30

$$\frac{\text{def.}_{(r=x)}}{\text{def.}_{(r=20)}} = \frac{x^{1.75}}{20}$$

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[3] J. T. MacKenzie, "A Note on the Effect of Span on the Results of the Transverse Test of Cast Iron," *Transactions*, Am. Foundrymen's Assn., Vol. IV, No. 4, October, 1933.

[4] C. H. Adamson and G. S. Bell, "Transverse and Other Tests on Cast Iron Bars," *Carnegie Scholarship Memoirs*, Vol. XVI, p. 1 (1927).

that is, deflection varies as $r^{1.75}$. By Eq. 2, however, where modulus of elasticity, modulus of rupture, and diameter are constant, deflection varies as r^2.

Therefore

$$\frac{\text{actual def.}}{\text{theoretical def.}} = \frac{r^{1.75}}{r^2}$$

Or, according to the data of Heller and Jungbluth,[2] if $r = 20$ is considered as the standard where actual and theoretical values conicide

$$\text{actual def. percentage of theoretical def.} = \left(\frac{r}{20}\right)^{-0.25} \times 100$$

This relation is plotted in Fig. 1 with the deflection data of MacKenzie,[3] and Adamson and Bell[4] expressed as actual deflection percentages of theoretical deflections for varying values of r.

The equation for the points from MacKenzie's data is approximately

$$\text{Percentage deflection} = \left(\frac{r - 3.59}{20 - 3.59}\right)^{-0.1}$$

TABLE I.—RECALCULATED RESULTS FOR 2.20-IN. DIAMETER BARS.

	Average Modulus of Rupture, lb. per sq. in.			Average Modulus of Elasticity, lb. per sq. in.		
	18-in. Span	24-in. Span	Difference	18-in. Span	24-in. Span	Difference
All bars (15 tests)	58 000	56 000	−2000	10 080 000	10 490 000	410 000
All bars—corrected for span	54 100	53 800	−300	11 200 000	11 160 000	−40 000

TABLE II.—0.4 BY 0.5-IN. HARDENED STEEL BAR STRESSED TO 50,000 TO 60,000 LB. PER SQ. IN.

Deflection measured as in Fig. 2.

	12-in. Span	8-in. Span	6-in. Span	4-in. Span	3-in. Span	2-in. Span
1. Ratio, span to depth	30	20	15	10	7.5	5
2. Load, lb	220	360	415	765	1072	1576
3. Measured deflection, in	0.0950	0.0475	0.0245	0.0150	0.0104	0.0076
4. Apparent modulus of elasticity, lb. per sq. in.	30 200 000	29 200 000	27 600 000	24 600 000	21 100 000	12 500 000
5. Load to give 70,000-lb. per sq. in. fiber stress, lb	320	480	640	960	1280	1930
6. Deflection (calculated) for loads (5) modulus of elasticity (4), in	0.138	0.0633	0.0376	0.0188	0.0122	0.0093
7. Deflection (calculated) for loads (5) to give modulus of elasticity of 30.2, in	0.138	0.0611	0.0344	0.0153	0.0086	0.0038
8. Line (6) − (7) = spurious deflection, in	0	0.0022	0.0032	0.0035	0.0036	0.0054

This curve is asymptotic to $r = 3.59$. However, it is considered that below $r = 10$, the deflection values are not true beam deformations, because the conditions approach that of a shear test. If three points from MacKenzie's data below $r = 10$ are disregarded, the curve asymptotic to $r = 0$ which fits the other points is found to be

$$\text{Percentage deflection} = \left(\frac{r}{20}\right)^{-0.1} \times 100$$

or, deflection varies as span to the 1.90 power instead of as span to the second power.

Modulus of Elasticity:

Modulus of elasticity is a function of both modulus of rupture and deflection, varying inversely as deflection and directly as modulus of rupture. Values for modulus of elasticity in per cent of the value of modulus of elasticity for $r = 20$ from the data of Heller and Jungbluth,[2] and MacKenzie[3] are also plotted in Fig. 1.

By the data of Heller and Jungbluth, modulus of elasticity varies as $r^{0.18}$.

By the data of MacKenzie, modulus of elasticity—when r is greater than 10—varies as $r^{0.03}$.

Therefore, these data indicate that, in order to make modulus of elasticity independent of span, the term representing length of span indicated as being raised to the second power in Eq. 2, should be indicated as being raised to the 2.18 power (Heller and Jungbluth) or to the 2.03 power (MacKenzie).

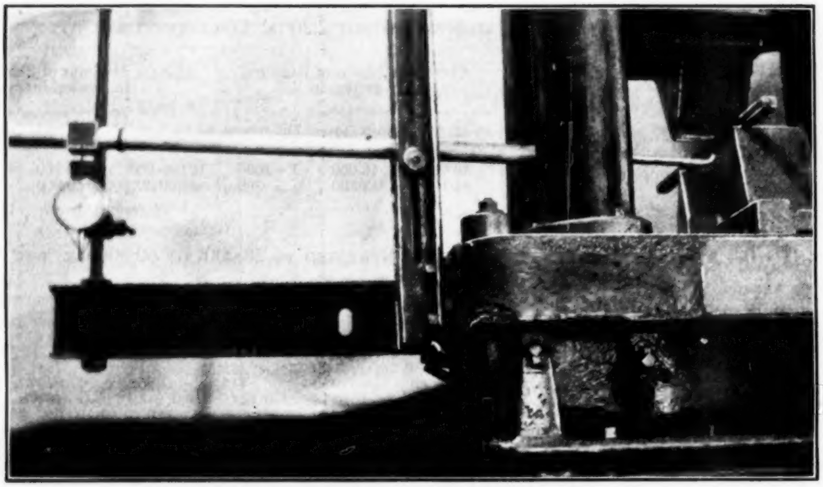

FIG. 2.—Regular Method for Deflection.

Dial actuated by aluminum beam mounted on knifeedge measures decrease in distance between bottom of bar and bed plate of testing machine.

Correction of Round Bars for Span:

The table below gives correction factors taken from the relations established for modulus of rupture and modulus of elasticity on varying spans. Dividing the values calculated by the conventional beam formulas by the proper factors gives in each case the value which would have been obtained if the bar had been broken on a span of 20 depths.

RATIO, SPAN TO DEPTH	MODULUS OF RUPTURE	MODULUS OF ELASTICITY
30	97	104
25	98	102
20	100	100
15	102	97
10	105	93
5	110	—

As a check and as an example of the use of these correction factors, the results of a number of tests on 2.20-in. diameter bars made by MacKenzie[5] on 18- and 24-in. supports were corrected for span, the recalculated results being given in Table I. A 2.20-in. bar on an 18-in. span is a span of 8 depths and on a 24-in. span is a span of 11 depths. Interpolating from the table of correction factors we have for spans of 8 and 11 depths:

	8 DEPTHS	11 DEPTHS
Approximate correction factor for modulus of rupture..............	107	104
Approximate correction factor for modulus of elasticity..............	90	94

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[5] J. T. MacKenzie, "Tests on Cast Iron Specimens of Various Diameters," *Proceedings*, Am. Soc. Testing Mats. Vol. 31, Part I, p. 160 (1931).

As may be seen, there is a definite difference between the average values from tests on the longer and on the shorter span. Modulus of rupture is larger on the shorter span and modulus of elasticity larger on the longer span. When these values are corrected for span, the differences become practically negligible.

It must be remembered that these correction factors represent only the data considered and are not necessarily true for all test conditions or for all irons.

RECTANGULAR BARS

The experimental work on the rectangular cast-iron bars involved a large number of remarkably uniform specimens and effort was made to insure the accuracy of the measurements. Though the same trends were found for the test results with varying spans, an hyperbolic equation was found to fit the experimentally determined points better than the parabolic equation used for round bars.

Calibration of Measurements:

In the tests on cast iron specimens the original deflection measurements were in each case the decrease in distance between the bottom of the bar under the load point and the bed-plate of the machine. These measurements were made by means of an aluminum beam mounted on a knife-edge with one end under the center of the bar and the other end resting on an Ames dial reading to 0.0001 in. mounted from the bed-plate of the testing machine (see Fig. 2).

In order to check the accuracy of these deflection measurements a hardened steel bar of the same section as the cast iron bars to be tested (0.4 by 0.5 in.) was stressed to 50,000 to 60,000 lb. per sq. in. ultimate fiber stress on each of the spans used. It was found that on the first loading the steel bar showed appreciable plastic set, that is, did not return to zero deflection upon removal of the load, so that only the third loading of the bar, on each span, was recorded. Results of these tests are shown in Table II.

Using the measured deflections the moduli of elasticity obtained on the same steel bar decreased rapidly with decreasing span. Measured deflection calculated to 70,000 lb. per sq. in. fiber stress less the calculated deflection to give the modulus of elasticity of 30,200,000 lb. per sq. in. was considered to represent the spurious deflection. (Of course, there is some spurious deflection in the test on a 12-in. span, from which the figure of 30,200,000 was obtained, but this was considered to be a negligible proportion of the measured deflection.)

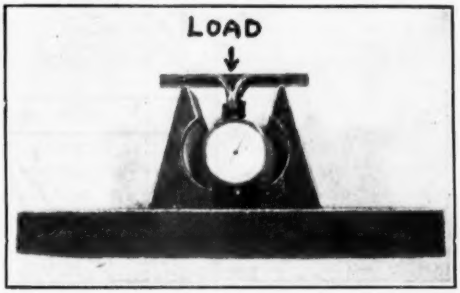


FIG. 3.—Method for Deflection Used in Calibration. Dial is hung by a yoke from neutral axis of bar above support points.

The above calculation of error in deflection readings is based on the assumption that the modulus of elasticity of the steel bar calculated by the ordinary beam formula is constant for the lengths of span investigated. This would seem to be theoretically untrue, as it is evident that as length of span decreases a proportionately larger part of the measured deflection will be due to shear strain instead of true beam deflection. In order to check this point an effort was made to measure in so far as possible the true deflection of the steel bar on the short spans, obviating previous deflection errors.

The Ames dial reading to 0.0001 in. was attached to a yoke hung from two $\frac{1}{16}$-in. round pins through the neutral axis of the bar exactly above the support points. The tip of the dial rested on the bottom of the bar at the center of the span and directly beneath the load point (Fig. 3). By this method, the crushing of the bar at the support points, the compression of the supports, etc., did not appear as measured deflection.

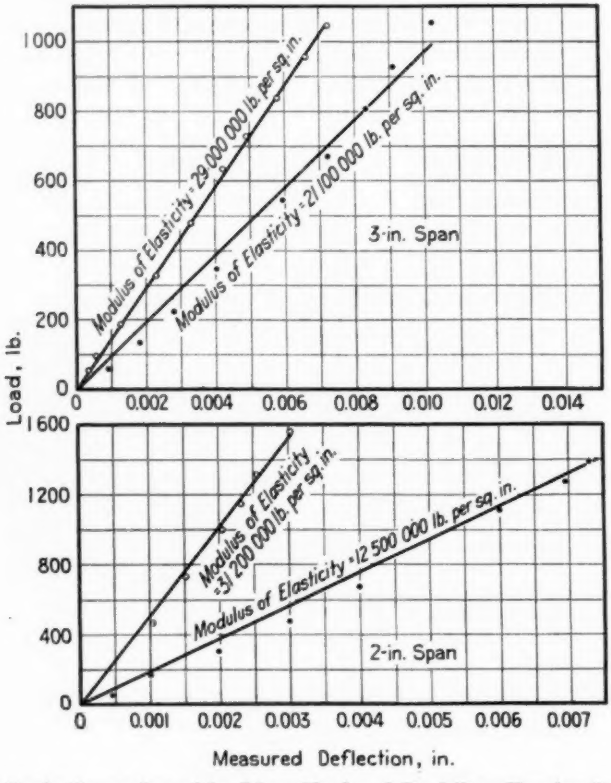


FIG. 4.—Stress-Strain Curves for a 4 by 5-in. Hardened Steel Bar, Showing Spurious Deflection Obtained in Ordinary Method of Measurement.

Solid Circles are from method shown in Fig. 2.
Open Circles are from method shown in Fig. 3.

This test gave deflection values much smaller than those of the previous method. Modulus of elasticity values for the same steel bar obtained by this method on both 3 and 2-in. supports were always within the range of 28,500,000 to 31,500,000 lb. per sq. in. (see Fig. 4).

Since this is a check with the 30,200,000 lb. per sq. in. modulus of elasticity found on the 12-in. span it would seem that the deflection due to shear, even on 2-in. span, is not enough to be detected by the test methods used, and that the values shown in Table II as spurious deflection are approximately correct.

Calibration of the weighing device for measuring loads showed the maximum error to be in the order of 1 per cent.

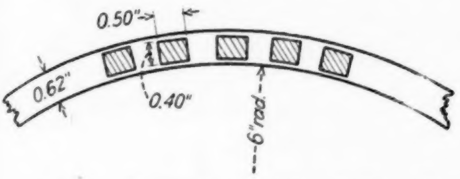


FIG. 5.—Segment of a Ring Section of a 12-in. Pipe.

Shaded portions show section of test specimens.

TABLE III.—SUMMARY OF TESTS ON MONO-CAST STRIPS.

	12-in. Span	8-in. Span	6-in. Span	4-in. Span	3-in. Span	2-in. Span
Ratio, span to depth	30	20	15	10	7.5	5
Number of tests	9	10	13	10	10	9
Average modulus of rupture, lb. per sq. in	68 200	67 600	68 200	68 500	69 700	72 800
Average deflection, in	0.3700	0.1648	0.0933	0.0456	0.0260	0.0138
Deflection corrected to 8-in. span, in	0.1642	0.1648	0.1660	0.1824	0.1850	0.2210
Average modulus of elasticity, lb. per sq. in	11 100 000	11 000 000	11 100 000	10 000 000	10 000 000	8 750 000

TABLE IV.—SUMMARY OF TESTS ON SUPER-DE LAVAUD STRIPS.

	12-in. Span	8-in. Span	6-in. Span	4-in. Span	3-in. Span	2-in. Span
Ratio, span to depth	30	20	15	10	7.5	5
Number of tests	9	9	8	10	10	9
Average modulus of rupture, lb. per sq. in	61 400	62 400	62 500	63 900	66 600	72 000
Average deflection, in	0.3970	0.1828	0.1039	0.0480	0.0348	0.0191
Deflection corrected to 8-in. span, in	0.1765	0.1828	0.1846	0.1920	0.2477	0.3057
Average modulus of elasticity, lb. per sq. in	9 300 000	9 100 000	9 000 000	8 860 000	7 200 000	6 300 000

Results on 0.4- by 0.5-in. Rectangular Cast-Iron Transverse Test Specimens:

From a 14-in. long ring section of a 12-in. class C mono-cast pipe were cut thirty-six ½-in. wide strips. The wall thickness of the pipe was 0.68 in., but after cutting, the strips were planed down to 0.40-in. thick, equal cuts being taken on the inside and outside surfaces (see Fig. 5).

The resulting strips, 0.40 by 0.50 in. in section, were broken on a 5000-lb. Rhiele universal testing machine in the transverse test with single point loading at the center. In each case that side of the strip originally nearest the outer surface of the pipe was the tension side. At least ten tests were made on each of the following spans: 12, 8, 6, 4, 3 and 2-in. Except for the 12-in. span specimens which were the full 14 in. long, the specimens for the other spans were cut to a length 1 in. greater than the span, so that there was little overhanging. No test was made on broken halves from another test.

Tests in which flaws were noted at the break, or in which faulty seating on the supports gave unreliable deflection values, were thrown out. Rate of movement of the loading head in all tests was 0.066 in. per min. In calculating the results shown in Table III, the ultimate deflection readings were corrected for the error in deflection as determined in the section above on Calibration of Measurements. Figure 6 shows these results graphically with the spread indicated by frequency curves about each average point. The deflection values plotted are corrected by the conven-

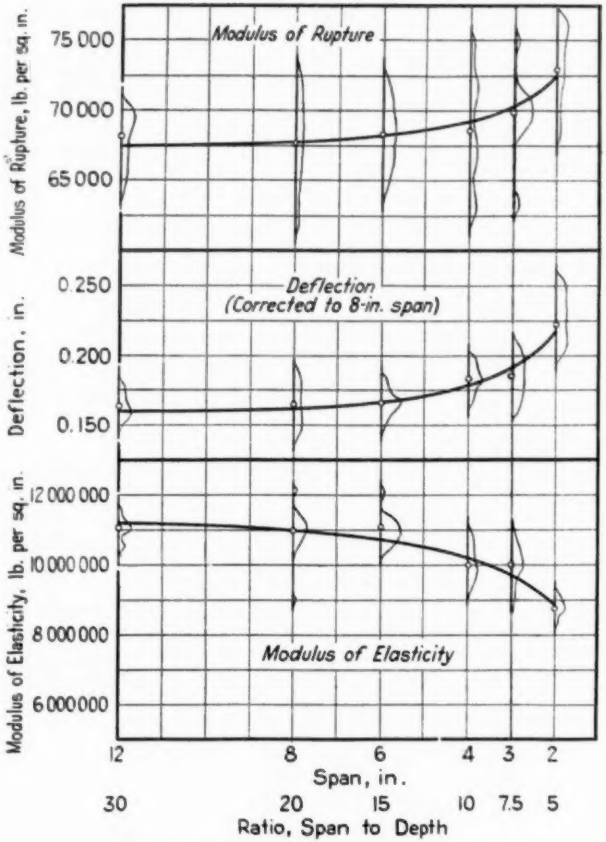


FIG. 6.—Strips from Mono-Cast Pipe.

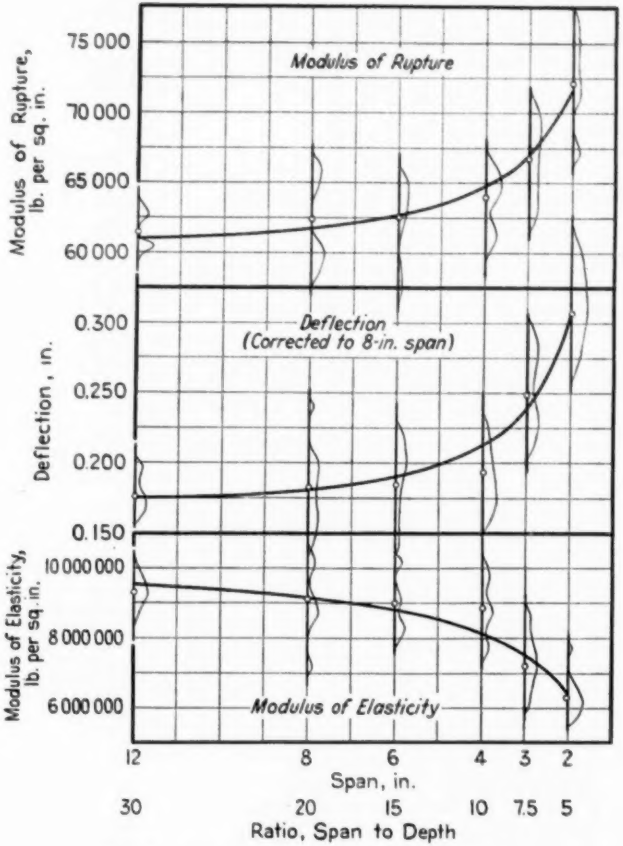
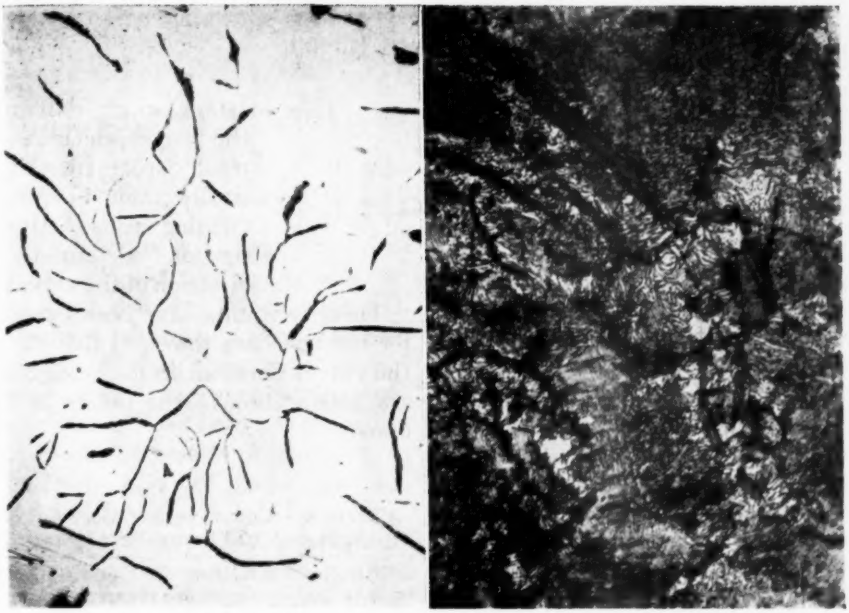


FIG. 7.—Strips from Super-de Lavaud Pipe.



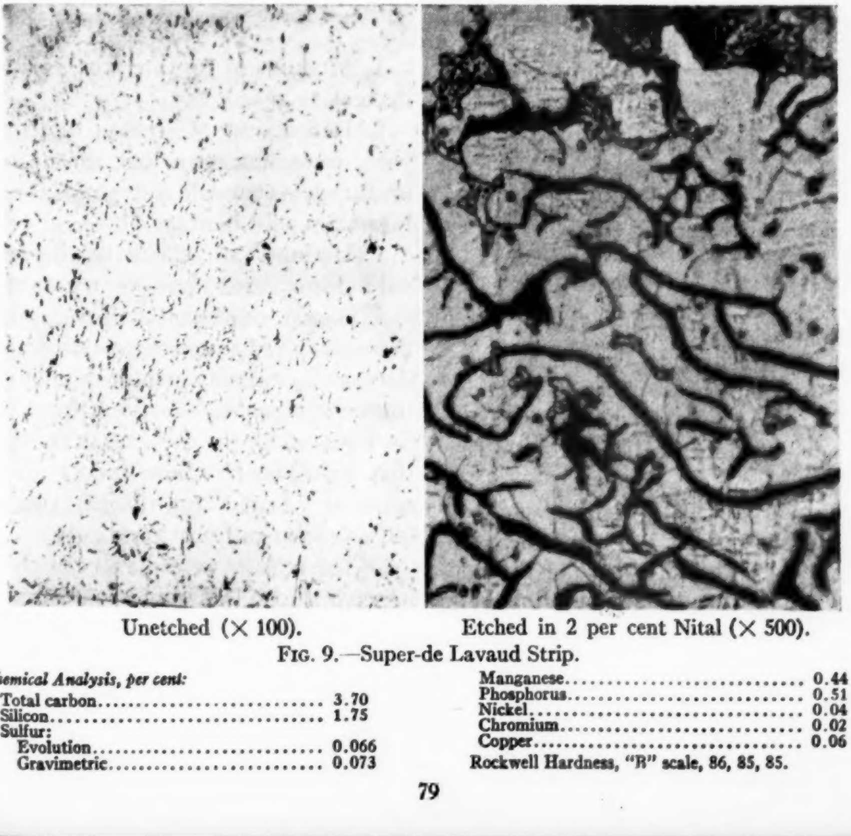
Unetched (× 100). Etched in 2 per cent Nital (× 500).

FIG. 8.—Mono-Cast Strip.

Chemical Analysis, per cent:

Total carbon	3.20
Silicon	1.54
Sulfur:	
Evolution	0.055
Gravimetric	0.064
Manganese	0.51
Phosphorus	0.62
Nickel	0.05
Chromium	0.02
Copper	0.04

Rockwell Hardness, "B" scale, 93, 91, 94.



Unetched (× 100). Etched in 2 per cent Nital (× 500).

FIG. 9.—Super-de Lavaud Strip.

Chemical Analysis, per cent:

Total carbon	3.70
Silicon	1.75
Sulfur:	
Evolution	0.066
Gravimetric	0.073
Manganese	0.44
Phosphorus	0.51
Nickel	0.04
Chromium	0.02
Copper	0.06

Rockwell Hardness, "B" scale, 86, 85, 85.

tional beam formula to a span of 8 in. by the equation

$$\text{Corrected deflection (8-in. span)} = \text{Deflection (span} = x)\,\frac{8^2}{x^2}$$

This was done in order to make the deflection results comparable, and so that the deviation of the points from a horizontal line represents the deviation of actual test measurements from the theoretical beam formula relations.

This series of tests was repeated for a different type of iron using strips cut from a 12-in. super-de Lavaud pipe of the same thickness. Table IV and Fig. 7 show the results obtained in the tests of these specimens.

In Figs. 8 and 9 are shown micrographs and analyses of the two irons tested. The mono-cast pipe is centrifugally cast against a sand-lined mold. The super-de Lavaud pipe is centrifugally cast against a water-cooled steel mold which has been coated with a pulverulent material and then annealed.

Mathematical Analysis:

The type of equation which seems to fit best the average points for the test values (Figs. 3 and 4) is an hyperbolic equation of the general type:

$$T = \frac{Ur}{r + C} \quad \text{. . . . . (5)}$$

where T = test results (modulus of rupture, corrected deflection, or modulus of elasticity),

r = length of span, expressed in depths $\left(\frac{\text{span}}{\text{depth}}\right)$,

U = constant representing the ultimate value of the test result for an infinitely long span, and

C = constant which determines the rate at which the test result departs from the ultimate value U with decreasing spans. Also the sign of C determines the direction of the curvature.

These constants have been evaluated for the test data shown (Table V), and the curves obtained from the equations are drawn through the points in Figs. 6 and 7.

Table V.—Constants for Eq. 5 Giving Curves to Fit the Test Data.

	Mono-Cast			Super-de Lavaud		
	Modulus of Rupture	Corrected Deflection	Modulus of Elasticity	Modulus of Rupture	Corrected Deflection	Modulus of Elasticity
U.	66.5	0.152	11.8	59.0	0.160	10.5
C.	−0.4	−1.5	1.6	−0.9	−2.4	3.0

CONCLUSIONS

1. Modulus of rupture increases with decreasing span.

2. Deflection, of course, decreases with decreasing span, but the decrease is much less than the conventional beam formula would indicate.

3. Modulus of elasticity decreases with decreasing span.

4. The rate of change of the values from these super-de Lavaud strips with decreasing span is generally greater than that of the values from mono-cast strips. This is evidenced by the C values in the equation used, which show the C for the super-de Lavaud test roughly twice as large (numerically) in each case.

5. The amount of the effect of changing span depends largely upon the properties of the iron.

DISCUSSION

Mr. H. W. Stuart[1] *(presented in written form).*—Messrs. MacKenzie and Donoho have presented a very interesting paper, in that they have shown what can be expected when testing cast iron on spans shorter than are generally used. Since the results obtained show practically no effect of span except for the 4, 3 and 2-in. spans, it is interesting to investigate the results of these short spans more closely. Although considerable effort was made to make these tests under uniform conditions, there was a great deal of difference in the speed of load application on the various spans. It is stated in this paper that the rate of movement of the loading head was 0.066 in. per min. in all tests. This being the case, actual load application was made at rates of approximately 50 lb. per min. on the 12-in. span and 9500 lb. per min. on the 2-in. span. This wide difference in rate of load application has two effects: (1) very rapid testing has a tendency to give higher results; (2) the same accuracy cannot be expected from the test machine at these widely variable speeds.

Aside from speed of testing there is, I believe, a very definite reason for higher rupture and lower elasticities being obtained on the very short spans. The beam formula is based on the fact that maximum stress occurs directly under the point of load application and decreases directly from this point to zero over the supports. We know that this is not strictly true and a considerable portion of metal adjacent to the center of the span is stressed to a point higher than it theoretically should be. As the span is changed, the portion of the beam which is stressed higher than it should be does not necessarily vary directly with the length of span. The results obtained by Messrs. MacKenzie and Donoho would indicate that on the very small spans a considerably greater portion of metal is stressed above that which it theoretically should be, and the stress distribution diagrams in the accompanying Fig. 1 show that such a result is to be expected. This metal which is stressed higher than it theoretically should be of course produces an increase in deflection and a consequent lower modulus of elasticity. The fact that the material having the lower modulus of elasticity on the longer spans changed a greater amount on the shorter spans is, I believe, very good evidence of this point.

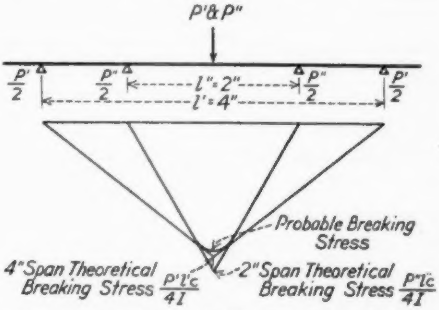

P'& P"
$\frac{P'}{2}$
$\frac{P''}{2}$
$l'' = 2''$
$\frac{P''}{2}$
$\frac{P'}{2}$
$l' = 4''$
Probable Breaking Stress
4" Span Theoretical Breaking Stress $\frac{P'l'c}{4I}$
2" Span Theoretical Breaking Stress $\frac{P''l''c}{4I}$

Fig. 1.—Variation in Theoretical Breaking Stress with Length of Span.

Conclusion 1—"Modulus of rupture increases with decreasing span" and Conclusion 2—"Modulus of elasticity

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[1] Research Dept., United States Pipe and Foundry Co., Burlington, N. J.

decreases with decreasing span" are limited by the data to the spans under 6 in., and with this as a basis it might be well to conclude further that transverse testing should be limited as far as possible to spans equal to at least fifteen times the height of the specimen being tested.

Mr. W. J. Schlick[2] (*presented in written form*).—The authors have presented the results of an interesting study, and have drawn conclusions in conformity with the data. However, it seems probable that the relationships shown between the span-depth ratio and the physical properties of the specimens may have resulted more from the method of test than from characteristics inherent in cast iron as a material.

If the loading conditions are such that the beam is in pure flexure at the location of the failure, and if the beams of different depths and spans are uniform in physical properties, the resisting moments (or resisting strengths) will vary with the depth, and will be independent of the span. If the beams be uniform in both cross-section and physical properties, as were those of each of the two series of rectangular beams (mono-cast and super de Lavaud), the resisting moment for each series should be constant. If the beams are of different depths and have the general elastic and strength properties of cast iron, the moduli of rupture may not vary directly with the actual resisting moments, since the relative positions of the actual and assumed neutral surfaces may not be constant.

If the beam be loaded at midspan, all transverse sections, except the section directly under the load, will be subject to both shearing and flexural stresses. The unit shears for beams of uniform cross-section will vary directly with the breaking loads, thus decreasing with increasing span. Thus, the moduli of rupture of cast iron beams testing with center loading may vary with both span and depth. However, if the beams be of uniform depth, as were the rectangular beams reported in this paper, variations in moduli of rupture would appear to be functions of span only, and not of the span-depth ratio.

It appears also that the method of loading used may have introduced some restraint at the load and the supports. This phase of the problem has a parallel in the development of the flexure test for plain concrete beams. A few years ago the materials laboratory of one of our state highway departments found that the moduli of rupture of concrete beams were varying with some function of the span-depth ratio. Before this relationship could be evaluated it was found that the variation in moduli was due, in part at least, to restraint at the supports.

About three years ago the Committee on Materials of the American Association of State Highway Officials sponsored an extensive study of the effects of span, width and depth upon the moduli of rupture of plain concrete beams. All of the beams were tested, with third-point loading, in an apparatus which removed practically all possible restraint at both loads and supports. The results of the tests showed that span and width had negligible effects, but that the moduli decreased from 860 lb. per sq. in. for beams 4 in. deep, to 760 lb. per sq. in. for beams 10 in. deep. However, there was one element of uncertainty which indicates that a portion of this reduction in modulus of rupture was due to the procedure followed, and not to characteristics inherent in concrete. Although the effect of this element of uncertainty cannot be evaluated numerically, it would seem that, with third-point

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[2] Civil Engineer, Engineering Experiment Station, Iowa State College, Ames, Iowa.

loading and removal of restraint at loads and supports, the variation in modulus of rupture with span and depth is small.

Unfortunately our knowledge of the action of cast iron under flexure necessitates that such problems be studied empirically. Messrs. MacKenzie and Donoho have presented data for one method of loading and one depth of beam. It would be interesting to see similar results for beams in which the depth was varied, and for beams tested with other conditions of load and support.

MR. J. B. KOMMERS[3] (*presented in written form*).—To those who have occasion to study tests of cast iron, this paper is of considerable interest, because the results show so definitely how the strength of the specimen is influenced by the ratio of the span to the depth of the bar.

The deflection results on the hardened steel bar indicate that in order to obtain accurate measures of deflection, the device used in measuring deflection should be supported from the neutral axis of the test bar immediately above the supports.

The practical conclusion to be drawn from these tests is that transverse specimens of cast iron must be standardized in order that comparable strength results may be obtained. Furthermore, if the deflection of cast iron beams is to be determined accurately, the technique used in measuring deflection should be standardized.

MR. M. O. WITHEY[4] (*presented in written form*).—The data presented on the effect of the ratio of span to depth on the modulus of rupture of rectangular cast iron bars are of value in making strength comparisons when tests of such material are conducted on specimens differing in dimensions.

The data labeled modulus of elasticity are misnamed. The application of this terminology should indicate that the load-deflection curves for these specimens were straight lines up to the breaking loads. From the data presented, this is evidently not the case. If this so-called "modulus of elasticity" has a definite value, would it not be better to name it the "stiffness factor"?

The experience of the authors serves to emphasize the importance of eliminating spurious deflections in transverse tests of brittle materials due to localized deformations at contacts and machine deformations.

It would seem that more informative data on cast iron would be obtained if, in general, the load-deflection curves were determined. By this procedure it would be possible to ascertain from a single test the modulus of elasticity, measuring stiffness at working stresses; the modulus of rupture, measuring strength; the energy of rupture (area under curve), measuring toughness; the maximum deflection, measuring flexibility; and the character of fracture.

MR. H. F. MOORE[5].—The authors are indeed to be commended for pointing out the serious errors which may arise from the measurement of deflections of beams by the use of apparatus which includes deflection of machine parts. At the University of Illinois it has frequently been found convenient to measure not the deflection of a beam, but the change of slope from end to end, or between two definite points along the beam. This change of slope can be measured by the motion of pointers attached directly to the beam, with some micrometer arrangement measuring the change of distance

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[3] Professor of Mechanics, Engineering College, University of Wisconsin, Madison, Wis.

[4] Professor of Mechanics, University of Wisconsin, Madison, Wis.

[5] Professor of Engineering Materials, University of Illinois, Urbana, Ill.

between the ends of the pointers, as is shown in the accompanying Fig. 2.

The strain along the extreme fibers of the beam, and the modulus of elasticity, can be determined from this change of slope as readily as from the deflection.

Might I suggest that, if modulus of elasticity is determined from the slope of a straight line from the origin of the stress-strain curve to any point on that curve, the term "secant modulus" be used, and if the modulus of elasticity is determined from the slope of the tangent at the chosen point on the stress-strain curve the term "tangent modulus" be used. This follows a practice rather common in concrete testing.

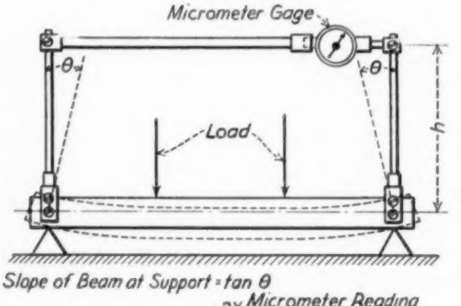
Micrometer Gage
θ
θ
Load
h
Slope of Beam at Support = tan θ
= 2X Micrometer Reading / 2h

FIG. 2.—Measuring Change of Slope.

MR. W. L. COLLINS[6].—During the past year a few tests of rectangular cast iron bars for determining the effect of span-depth ratio on the modulus of rupture in bending were made at the University of Illinois. The tests were entirely of an exploratory nature and the only definite conclusion reached was that the problem was one requiring further study.

From the tests that were expected to give the best results some differences from the findings of Messrs. MacKenzie and Donoho were noted. In the low range of span-depth ratios the value of modulus of rupture did not decrease rapidly and for the longer span-depth ratios some surprisingly high values were found.

MR. J. E. HURST[7] (*by letter*).—The conventional beam formulas for modulus of rupture, elasticity, and deflection are based upon a mathematical consideration of the case of "simple bending" in which one of the important assumptions made is that shearing forces are absent. Not only are these conventional formulas known to be incorrect for cast iron, but they are of course of an approximate charactei only for all materials.

Numerous attempts have been made to incorporate the deviations from "simple bending" into mathematical considerations of bending with the object of obtaining relationships which shall more nearly represent the results obtained by experimental methods. Relationships of logarithmic and exponential character have been produced from such considerations, which bear comparison with expressions derived from experimental results such as those shown by the authors. The fundamental difficulty always remains that the magnitude of deviation of actual test measurements from the theoretical or mathematically derived beam formula relations is always found to depend largely on the qualities of the material. This is particularly true in the case of cast iron and is very clearly demonstrated by the authors in their experimental results obtained from samples of "Sand Spun" and "Metal Mould" centrifugally cast material. In the case of these specimens the constant related to the effect of decreasing span differs roughly about 100 per cent in the two materials. The authors would no doubt agree that materials produced by the same processes but of different

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[6] Instructor, Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[7] Lichfield, Staffordshire, England.

compositions would not of necessity yield results similar in magnitude to those reported for the particular specimens examined by them.

The tests summarized in Tables III and IV were conducted on specimens obtained from a single pipe in each case and my attention is attracted by the degree of "scatter" of the results about each average point. This in itself is an illuminating comment on the difficulty of formulating mathematical relationships, conventional or otherwise, but it also indicates the direction of further investigation work.

MR. J. G. PEARCE[8] (*by letter*).—Data supporting the author's first three conclusions may be found in my papers to the Iron and Steel Institute, 1928, Vol. II, p. 73 and 1934, Vol. I, p. 331, and a note on span influence on transverse strength was given in the *Bulletin* of the British Cast Iron Research Association, 1929, p. 97. I have examined much experimental data with a view to evolving mathematical relations covering the effect of span changes on rupture modulus (which in Britain we now prefer to call transverse rupture stress), deflection and elastic modulus, but have always concluded that the experimental terms involved vary from iron to iron, and am apprehensive that figures published for particular irons may be applied to others. The most satisfactory method is, therefore, to work on a standard span for a given size of bar.

In view of the admitted inaccuracy of using the total deflection figure in such equations as Eqs. 2 and 4 of the present paper, in the second of the above-mentioned papers I separated the plastic component from the total deflection, a quite simple procedure, and suggested that the difference, conveniently termed elastic deflection, forms a more accurate basis for the deflection figure in such calculations. Elastic modulus results between different irons become more consistent, for elastic deflection figures depend on the matrix, and do not differ widely from iron to iron. The plastic deflections, however, vary very widely. A brittle iron (high in silicon or phosphorus) has a low, and a tough iron a high plastic deflection or permanent set. The high deflection of austenitic irons is due to high plastic deflection, and treatment in this way makes them consistent with other irons in regard to elastic modulus, while use of total deflection gives an absurdly low figure. Further experience has confirmed that many elastic deflection graphs bend slightly to the deflection axis, but not sufficiently to preclude treatment on the lines suggested, and the general results have been confirmed by Harbach, *Foundry Trade Journal*, August 20, 1936.

MESSRS. H. JUNGBLUTH[9] AND P. A. HELLER[9] (*by letter*) (*translation of discussion submitted by letter*).—The findings by MacKenzie and Donoho on the dependence of the bending strength (σ'_B) and the deflection (F_B) on the span-depth ratio ($\lambda = l_{s/d}$) are in excellent agreement with those of our own work. This is especially true in the range $\lambda = 10$ to $\lambda = 30$. The authors show truly that under $\lambda = 10$ not only bending stresses but difficultly evaluable shear stresses come into the problem, so that the observed values are no longer the properties of the material but of the test conditions.

It is not quite clear on what grounds the authors have chosen the exponent -0.07 for bending strength. It appears to us that our value of -0.105 is nearer the truth. The difference is indeed

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[8] Director, British Cast Iron Research Assn., Birmingham, England.

[9] Fried Krupp Aktiengesellschaft, Essen, Germany.

small but still the values given in Table I show that −0.105 gives a closer approximation:

	$MR_{(r=x)} = MR_{(r=20)} \cdot \left(\frac{x}{20}\right)^{-0.07}$			$MR_{(r=x)} = MR_{(r=20)} \cdot \left(\frac{x}{20}\right)^{-0.105}$		
	18 in.	24 in.	Difference	18 in.	24 in.	Difference
Observed....	58 000	56 000	−2000	58 000	56 000	−2000
Calculated...	54 100	53 700	−400	52 700	52 500	−200

This example shows chiefly that the equations are of importance for conversion of test results on different spans, chiefly when evaluating the work of foreign investigators. We think it would be helpful to prepare tables for the conversion of American to German tests (1.2 by 18 in. to 30 mm. by 600 mm.) and *vice versa*. A suggested table follows:

Form of Test Bar		German Test Bar	American Test Bar	
		30 mm. by 600 mm.	1.2 in. by 12 in.	1.2 in. by 18 in.
German	30 mm. by 600 mm.		kg. per sq. mm. × 87.0 = pounds	kg. per sq. mm. × 55.3 = pounds
			mm. × 0.0117 = inches	mm. × 0.0243 = inches
American	1.2 in. by 12 in.	pounds × 0.0115 = kg. per sq. mm.		pounds × 0.96 = pounds
		inches × 84.0 = mm.		inches × 2.04 = inches
	1.2 in. by 18 in.	pounds × 0.0181 = kg. per sq. mm.	pounds × 1.06 = pounds	
		inches × 41.2 = mm.	inches × 0.48 = inches	

MR. J. T. MACKENZIE[10] (*author's closure*).—This work was not intended to develop any theory. It was first developed as a report to the Flexure Testing Section of the Society's Committee E-1 on Methods of Testing, and was simply designed to show the order of error that may be encountered with the ordinary setup for deflection. One reason we went to these machined specimens is that it is very difficult to get a rectangular bar truly flat and have it lie down on the support so that a cast flat bar is really not reliable for such measurements. The idea of course was to show the point that whenever you are measuring deflections which depend on the relative motion of the bottom of the bar and the bed of the testing machine or the head of the cross-head, which is much worse, that you must look out for very large errors in deflection. They do not become important on long spans. But on something like the Fremont test specimen, which is 30 mm. span, and 10 mm. deep, or a ratio of span to depth of three, which is well down in the range which we investigated, it is quite apparent that no accuracy is possible unless it is done by some scheme such as Moore proposes or the scheme we are using. We discovered when Talbot was making his tests at the University of Illinois the misuse of the term modulus of elasticity. This has been apologized for so much we thought it would be trite to repeat the apology, but "stiffness factor" might be a good word; still the formula which we use to determine it is given in all the engineering and mathematical text books as the formula connecting load and deflection; so, with full knowledge of what we are doing, we use the words "modulus of elasticity." I do not think that Moore would seriously quarrel with us about continuing that terminology. As to Mr. Schlick's statement that there is only one test, he

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[10] Metallurgist, American Cast Iron Pipe Co., Birmingham, Ala.

overlooked the first half of the paper which reports tests on bars up to 2.2 in. in diameter. Jungbluth and Heller used 1.2 and Adamson and Bell used a range from 0.75 up to quite large bars, and so, while the rectangular results are only on a 0.4 in. specimen, it is not true that the paper as a whole is on only one beam.

When we go to very long spans, we get a curve beam problem which interferes with the ordinary calculations. As an illustration, we cut a ½-in. strip out of a 16-ft. pipe and tested it as a beam 15 ft. long. This was 0.34 in. in thickness and when suspended by wires from the ceiling, so as to allow the ends to move freely, the deflection due to its own weight was 30 in. or a calculated modulus of elasticity of 10,000,000 lb. It broke with a deflection of 50 in., having pulled in 21 in. from the sides. When this is calculated on a conventional beam formula on the original span it gives 82,500 lb. modulus of rupture with a modulus of elasticity of 30,000,000 lb. Pieces of this broken in the same way on normal spans of 10 in. gave a modulus of rupture of 48,000 lb. and a modulus of elasticity of 9,000,000 lb. Two pieces of the same strip tested on 60-in. spans with center loading broke with a modulus of rupture of 46,000 lb. and with a modulus of elasticity of 11,000,000 lb. This shows clearly that the transverse test must be held within reasonable limits in both directions.

In regard to the question raised by Messrs. Jungbluth and Heller that the exponent of the equation to fit the points for modulus of rupture in Fig. 1 should be −0.105 rather than −0.07 the authors are in some doubt as to what is meant. If only the three points from the data of Messrs. Jungbluth and Heller (*loc. cit.*) are considered, the equation

$$\text{Percentage value} = \left(\frac{r}{20}\right)^{-0.07} \times 100$$

fits better than the same equation with an exponent of −0.105 as the following table shows:

Ratio, span to depth	10	20	30
MR in percentage of value for ratio of span to depth = 20	105	100	97
Values obtained from percentage = $\left(\frac{r}{20}\right)^{-0.07} \times 100$	105	100	97.5
Values obtained from percentage = $\left(\frac{r}{20}\right)^{-0.105} \times 100$	107.5	100	96

If MacKenzie's points as plotted in Fig. 1 are also considered the difference in the goodness of fit is even more marked.

THE TENSILE STRENGTH OF CAST IRON

By J. O. Draffin[1] and W. L. Collins[1]

Synopsis

A report is presented on tests made to study the influence of surface defects on specimens held in flat and V-notch grips; on threaded-end specimens, both solid and hollow, to determine the effect of the interior portion on the distribution of stress over the cross-section; and on specimens with ends threaded on the outside and on the inside to study the effect of the location of threads on the strength of the specimen. Strain-gage readings of longitudinal deformation or strain were taken at small increments of load from zero to rupture on six gage lines spaced equally around the circumference of the specimen. Tests were made with both axial and eccentric loads.

Abrupt decreases in section appear to weaken the specimen more than surface defects. Relatively little difference was found between the strength of solid and hollow specimens, though the solid ones were slightly stronger. There was practically no difference between the strength of specimens with outside and inside threads. Attempts to produce eccentricity of loading by wedging the spherical bearing gripping device were successful at low loads but as the loads were increased the device adjusted itself so as to relieve the bending. Tensile strengths were approximately as great under eccentric loads as under axial loads. Specimens were unloaded from different stresses and reloaded to failure and this unloading and reloading had no apparent effect on the tensile properties. Permanent set after rupture was measured in a number of cases and was found to be about 40 to 50 per cent of the total deformation at rupture.

In many cases a knowledge of the tensile properties of cast iron is of considerable importance. This tensile strength is usually found by testing specimens of various sizes and shapes which are held in various ways; consequently there is a question as to the correctness of the results. This uncertainty regarding strength properties arises partly from the fact that for any given pouring they often vary within rather wide limits. Such variations are commonly attributed to one or more of three reasons, namely, (1) unaccountable differences in the quality of the metal, (2) localized stresses caused by internal discontinuities, surface defects, or abrupt changes in cross-section, and (3) accidental eccentricity of loading.

The data reported in this paper were obtained as a part of a larger investigation of the mechanical properties of cast iron which the authors are carrying on in the Engineering Experiment Station of the University of Illinois. In the course of the investigation it became evident that the actual tensile properties of the material, such as ultimate strength and unit strain or deformation at rupture, had to be determined. This re-

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[1] Professor, and Instructor, respectively, Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

quirement involved a knowledge of the distribution of the longitudinal strain on different sides of the specimen and the influence upon the strength and strain of the kind of specimen used, the method of holding it, and the effect of slight eccentricities. It was also desired to study the influence of surface conditions on the tensile properties.

To obtain information on the points stated above, two types of test specimens were used, a small round-hexagonal specimen, tested mainly in the as-cast condition, for the study of surface conditions, and a carefully machined round specimen to study other variations. The second type of specimen was used to investigate the following: (1) the distribution of the longitudinal deformation around the circumference of the specimen, studied by taking measurements on six longitudinal gage lines spaced 60 deg. apart; (2) the effect of the inner portion of the specimen on stress-strain relationships, studied by testing specimens which were solid and companion specimens in which the central portion had been drilled out, and (3) the effect on tensile strength of the method of applying load, studied by using specimens with threaded ends, some with the threads on the outside and companion specimens with the threads on the inside, and also by applying the load eccentrically to some of the specimens.

Acknowledgment is made to Prof. H. F. Moore for suggestions on the type of specimens used and to Prof. C. H. Casberg and Mr. J. F. Wooddell of the Machine Laboratories for supervising the making of the castings.

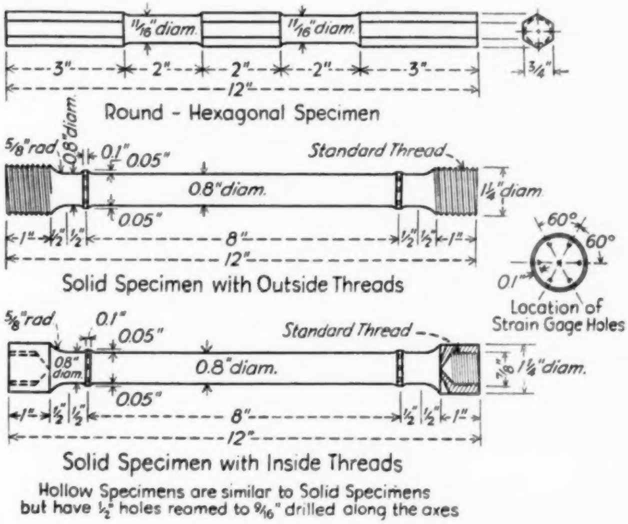

FIG. 1.—Test Specimens.

MATERIALS AND SPECIMENS

Round-Hexagonal Specimens.—The specimens used to determine the effect of surface conditions were of a good grade of cast iron purchased from a commercial foundry. They were taken from a stock of specimens used in the undergraduate laboratory work and were of the dimensions shown in Fig. 1. Thirty-six specimens were selected for testing; twelve had the central portion, that is the two circular and middle hexagonal portions, turned down in a lathe making a smooth, circular machined surface 6 in. long; twelve had as smooth surfaces as could be found; the remaining twelve were given surface defects by drilling 8 or 10 holes about 0.05 in. in diameter and 0.05 to 0.10 in. deep at various places on the two circular unmachined portions. While the small holes produced definite discontinuities in the surface, they did not reduce the area of the cross-section to an extent that the reduction needed to be considered.

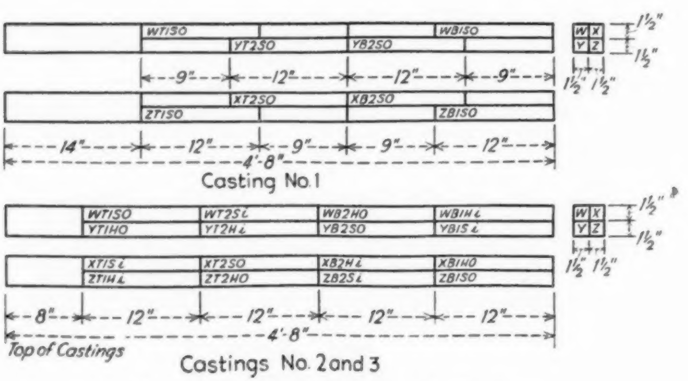

FIG. 2.—Position of Specimens in Castings.

Threaded-End Specimens.—The specimens with threaded ends, on which strain-gage readings were taken, were cut from three castings, poured from a single heat. The castings were 3 in. square and 56 in. long and were cast on end in the Machine Laboratories of the University of Illinois. The charge consisted of new pig, scrap cast iron and scrap steel, one-third of each. The chemical composition was calculated as approximately: total carbon 3.45 per cent, silicon 1.60 per cent, manganese 0.55 per cent, sulfur 0.09 per cent, phosphorus 0.45 per cent.

At the time the castings were made, six arbitration test bars 1.20 in. in diameter and 20 in. long were poured. These were broken on a span of 18 in. and the properties were as follows: modulus of rupture, 61,700 to 65,100 lb. per sq. in., with an average of 63,600 lb. per sq. in.; modulus of elasticity, as determined from load-deflection diagrams, 14,000,000 to 16,500,000 lb. per sq. in.

The location of each specimen in the casting is shown in Fig. 2 where the type of specimen, solid or hollow and inside or outside threaded ends, is indicated by a code. For instance, the specimen from casting No. 1, X quarter, upper part central portion, solid with outside threads, is marked 1XT2SO. The specimens were turned to the dimensions shown in Fig. 1 and were finished with a fine cut but were not polished; all work was done by one man to secure as uniform specimens as possible. In order that there might be no localized stress from strain-gage holes, a collar (see Fig. 1) in which strain-gage holes were drilled, was left on each specimen. This collar was small and was so far from the fillet at the change of section that it would not exert an appreciable effect on the strength of the specimen, which assumption proved to be justified.

METHOD OF TESTING

All specimens were tested in a 50,000-lb. screw-power testing machine. Some of the round-hexagonal specimens were tested with flat and some with V-notch grips.

The load was applied to all the threaded-end specimens by means of a bolt or pin on one end of which was a spherical head which rested in a spherical bearing block. The other end of the bolt or pin was fastened to the test specimen by means of a threaded sleeve. The specimens with outside threads were screwed directly into the threaded sleeve; the specimens with inside threads were connected to the sleeve by a stud bolt. Considerable care was used to adjust the loading pins for the first few tests in order to obtain an axial load; as the tests progressed the special precautions were seen to be unnecessary and in later tests no special care was taken to adjust the loading pins.

A double set of strain-gage readings, with check readings where needed, was taken for each increment of load, which was about 2000 lb. per sq. in. in the earlier stages of each test and reduced as the ultimate strength was approached. An effort was made to secure data for the stress-strain curves as near to rupture as possible and the results were most satisfactory since readings were obtained in a number of cases at the maximum load. In some tests the specimen broke while readings were being taken and in others the stress at rupture was only 200 or 300 lb. per sq. in. greater than that at the last strain-gage reading.

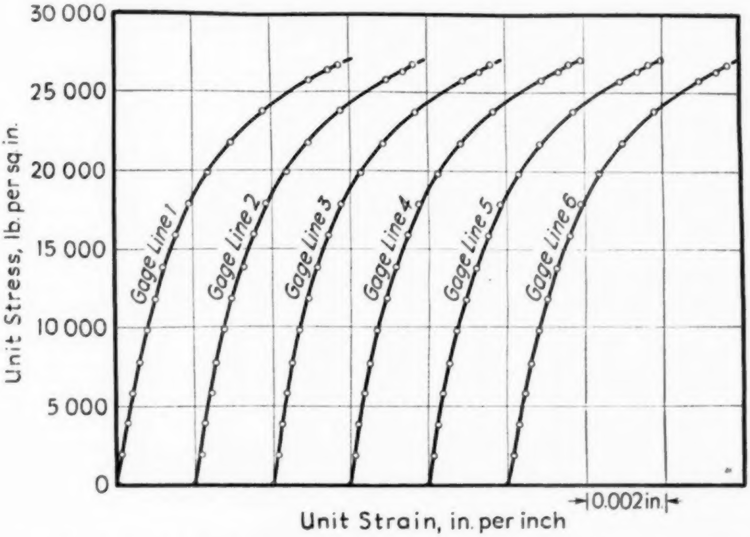

FIG. 3.—Stress-Strain Curves for Individual Gage Lines (Specimen 3XB2Hi).

TEST DATA

Only the ultimate load was obtained for the round-hexagonal specimens. The average ultimate strength for the twelve specimens of each type was as follows: as cast, smooth surface, 23,600 lb. per sq. in.; as cast but with artificial surface defects 24,700 lb. per sq. in.; machined round for 6 in. between hexagonal ends, 22,200 lb. per sq. in. The maximum percentage variation from the average for each type was 8.0, 11.0, and 15.0, respectively.

From the strain-gage data for the threaded-end specimens a curve was plotted for each gage line. A typical set of such curves, specimen 3XB2Hi, is shown in Fig. 3. These curves for the individual gage lines were plotted in order to study the distribution of longitudinal deformation around the circumference of the specimen and to detect any changes which might occur in the rate at which the different gage lines deformed. The average unit strain for all six gage lines was then taken for each increment of load and plotted as a composite curve. Typical composite curves for each type of specimen, solid with outside threads, solid with inside threads, hollow with outside threads and hollow with inside threads, are shown in Fig. 4.

Where the last deformation readings on any specimen were taken below the ultimate stress, the curve was extrapolated to that stress; portions of curves so extrapolated are shown with dotted lines. Readings were taken on two of the gage lines shown in Fig. 3 at the ultimate load, but the specimen broke before readings could be taken on the other gage lines at this load. A sufficient number of readings was taken at or near the ultimate load to give assurance that the shape of the extrapolated portion of the curve is substantially correct.

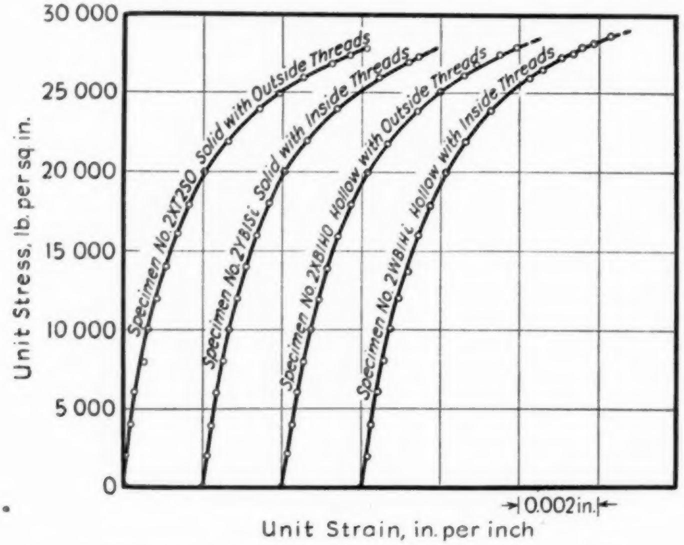

FIG. 4. Composite Stress-Strain Curves for Each Type of Specimen.

Table I gives data for each specimen on tensile strength, modulus of elasticity, and maximum average unit strain for the specimen as well as the maximum and minimum unit strains for any gage line on each specimen.

DISCUSSION OF DATA

Effect of Surface Discontinuities.—The tensile strengths of the round-hexagonal specimens show clearly that the small drill holes did not weaken the specimens though failure occurred in seven of the twelve at a drill hole. All but one of the other specimens, both machined and unmachined, broke at the junction of the circular and one of the end hexagonal portions. The conclusion must be drawn from these tests that slight surface defects do not affect appreciably the strength of the part but they tend to locate the place where the rupture will occur. While no quantitative evidence can be cited, the observed behavior of the test specimens suggests that the greatest weakening effect is where the end hexagonal portions join the circular portions. This may be the reason for the machined

TABLE I.—RESULTS OF TENSION TESTS OF CAST IRON.
Nominal diameter of specimens, 0.80 in. outside, 0.56 in. inside.

Specimen	Tensile Strength, lb. per sq. in.	Longitudinal Strain at Rupture, in. per inch			Modulus of Elasticity, lb. per sq. in.
		Single Gage Line		Average for All Gage Lines	
		Maximum	Minimum		
SOLID WITH OUTSIDE THREADS					
No. 1WT1SO	31 200	0.00685	0.00635	0.00664	15 500 000
No. 1YT2SO	31 400	0.00670	0.00636	0.00656	16 000 000
No. 1ZT1SO	30 200	0.00600	0.00485	0.00543	15 000 000
No. 1XT2SO	31 000	0.00640	0.00600	0.00625	17 000 000
No. 1WB1SO	30 700	0.00710	0.00680	0.00698	14 000 000
No. 1YB2SO	29 900	0.00715	0.00625	0.00665	15 000 000
No. 1ZB1SO	31 300	0.00687	0.00652	0.00668	14 000 000
No. 1XB2SO	30 900	0.00625	0.00495	0.00580	14 600 000
No. 2WT1SO	27 900	0.00617	0.00517	0.00567	14 700 000
No. 2XT2SO	27 800	0.00640	0.00585	0.00615	12 500 000
No. 2YB2SO	28 700	0.00720	0.00670	0.00696	14 500 000
No. 2ZB1SO	28 100	0.00610	0.00540	0.00574	12 400 000
No. 3WT1SO	29 000	0.00660	0.00648	0.00656	13 000 000
No. 3XT2SO	27 700	0.00653	0.00625	0.00636	12 800 000[b]
No. 3YB2SO	28 400	0.00700	0.00645	0.00677	13 800 000
No. 3ZB1SO	27 800	0.00620	0.00585	0.00605	11 100 000
SOLID WITH INSIDE THREADS					
No. 2WT2Si	27 800	0.00665	0.00620	0.00637	12 200 000[a]
No. 2XT1Si	27 700	0.00610	0.00540	0.00569	13 200 000[a]
No. 2YB1Si	27 800	0.00590	0.00570	0.00584	13 800 000
No. 2ZB2Si	28 500	0.00711	0.00680	0.00694	12 800 000
No. 3WT2Si	28 500	0.00715	0.00660	0.00684	13 100 000
No. 3XT1Si	28 700	0.00625	0.00595	0.00607	13 700 000
No. 3YB1Si	29 000	0.00720	0.00680	0.00704	12 700 000
No. 3ZB2Si	27 900	0.00710	0.00650	0.00672	12 500 000
HOLLOW WITH OUTSIDE THREADS					
No. 2YT1HO	26 400	0.00545	0.00500	0.00518	11 000 000
No. 2ZT2HO	27 800	0.00601	0.00542	0.00574	13 700 000
No. 2WB2HO	27 900	0.00650	0.00615	0.00636	12 200 000
No. 2XB1HO	28 400	0.00690	0.00630	0.00654	12 500 000
No. 3YT1HO	27 300	0.00565	0.00540	0.00555	12 300 000
No. 3ZT2HO	27 300	0.00625	0.00536	0.00573	13 500 000
No. 3WB2HO	27 900	0.00615	0.00575	0.00593	12 200 000
No. 3XB1HO	27 600	0.00550	0.00480	0.00518	12 000 000
HOLLOW WITH INSIDE THREADS					
No. 2YT2Hi	26 100	0.00476	0.00434	0.00456	14 000 000[a]
No. 2ZT1Hi	27 600	0.00595	0.00500	0.00548	13 400 000
No. 2WB1Hi	28 900	0.00700	0.00665	0.00678	13 000 000
No. 2XB2Hi	25 000	0.00485	0.00333	0.00406	12 000 000[a]
No. 3YT2Hi	25 900	0.00533	0.00467	0.00501	12 100 000[a]
No. 3ZT1Hi	26 700	0.00527	0.00464	0.00500	14 000 000
No. 3WB1Hi	28 000	0.00605	0.00590	0.00598	13 200 000
No. 3XB2Hi	27 100	0.00605	0.00590	0.00593	11 400 000

[a] Loaded eccentrically.

[b] Loaded eccentrically through Robertson shackles and broke in threaded end at load of 26,900 lb. per sq. in. Specimen put back in machine and loaded axially to failure.

specimens having the lowest strength of the three groups, since the change in section was greater for these pieces than for the others. No reason can be assigned for the highest strength being obtained from specimens with holes in them.

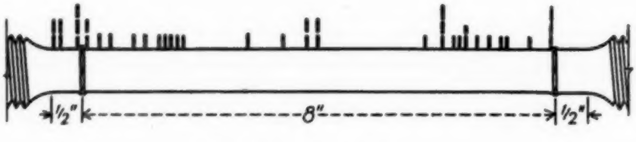

FIG. 5.—Location of Fractures.

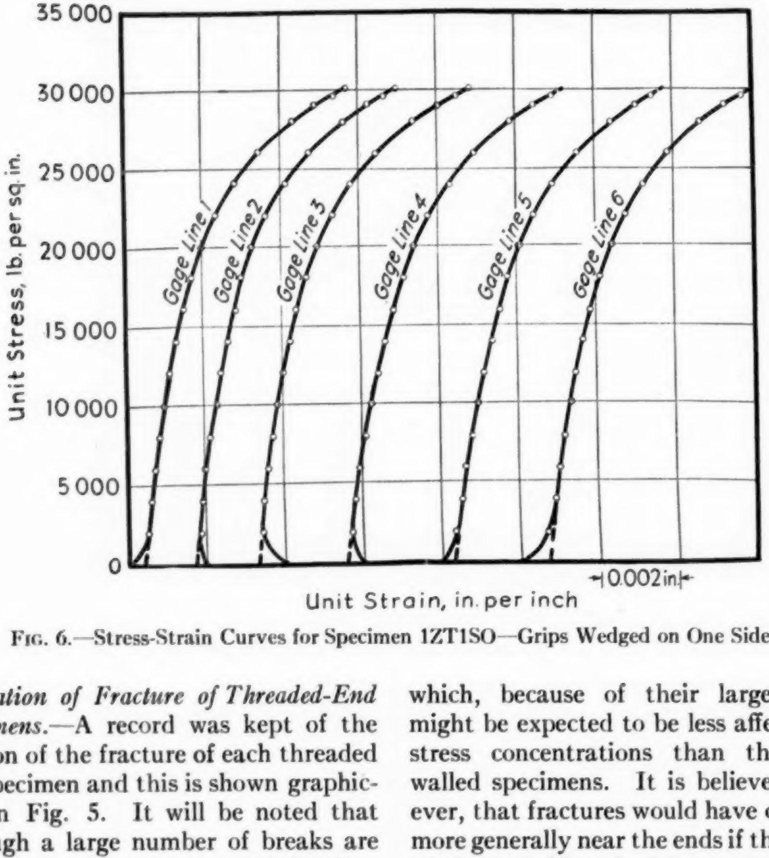

FIG. 6.—Stress-Strain Curves for Specimen 1ZT1SO—Grips Wedged on One Side.

Location of Fracture of Threaded-End Specimens.—A record was kept of the location of the fracture of each threaded end specimen and this is shown graphically in Fig. 5. It will be noted that although a large number of breaks are near one end, there is a general distribution of the breaks over the entire length of the reduced section, showing that the collars left on the specimens for strain-gage readings did not produce noticeable stress concentrations. Further evidence that failure was not due to stress concentration, either from fillets or collars, is furnished by the fact that most of the fractures at changes in section were in the solid specimens which, because of their larger area, might be expected to be less affected by stress concentrations than the thin-walled specimens. It is believed, however, that fractures would have occurred more generally near the ends if the radius of the end fillets had been appreciably less than the one used.

The location of fractures was not influenced by lateral movement of the crosshead of the testing machine as practically one-half the specimens broke above and one-half below the center line.

Comparison of Threaded-End Specimens.—The data given in Table I are averaged and assembled in Table II according to the different variables. Studies and comparisons of the data in Tables I and II show that two similar specimens taken from the same end of a casting, as 1WT1SO and 1ZT1SO, and tested in precisely the same manner may vary 1000 lb. per sq. in. in tensile strength. The maximum variation for individual specimens taken from different positions in different castings and tested in different ways is from a minimum of 25,000 lb. per sq. in. for specimen 2XB2Hi to a maximum of 31,400 lb. per sq. in. for specimen 1YT2SO. The average strength of casting No. 1 is about 10 to 11 per cent higher than the average strength of castings Nos. 2 and 3. The average tensile strength of the specimens, all tested in the same manner, taken from the top of castings is slightly less than the average tensile strength of specimens from the bottom of castings, with the exception of the solid specimens with outside threads from casting No. 3. The maximum variation is 7 to 8 per cent. The strengths of specimens intermediate between top and bottom do not vary in any regular way. Solid specimens averaged 2 to 5 per cent stronger than hollow ones, and specimens with outside threads averaged about 2 per cent stronger than those with inside threads. The solid specimens with outside threads gave, on the average, the highest strength of any combination.

TABLE II.—SUMMARY OF DATA FOR ESTIMATE OF EFFECT OF DIFFERENT VARIABLES OF TENSION TESTS.

Casting	Position of Specimen in Casting	Type of Specimen							
		Solid, Outside Threads		Solid, Inside Threads		Hollow, Outside Threads		Hollow, Inside Threads	
		Tensile Strength, lb. per sq. in.	Unit Strain at Rupture, in. per inch	Tensile Strength, lb. per sq. in.	Unit Strain at Rupture, in. per inch	Tensile Strength, lb. per sq. in.	Unit Strain at Rupture, in. per inch	Tensile Strength, lb. per sq. in.	Unit Strain at Rupture, in. per inch
No. 1	Top Quarter	30 700	....	....	....	....	....	....	....
	Bottom Quarter	31 000	....	....	....	....	....	....	....
	Average for Casting	30 800	0.00637	....	....	....	....	....	....
No. 2	Top Quarter	27 900	....	27 700	....	26 400	....	27 600	....
	Bottom Quarter	28 100	....	27 800	....	28 400	....	28 900	....
	Average for Casting	28 100	0.00613	27 900	0.00621	27 600	0.00595	26 900	0.00522
No. 3	Top Quarter	29 000	....	28 700	....	27 300	....	26 700	....
	Bottom Quarter	27 800	....	29 000	....	27 600	....	28 000	....
	Average for Casting	28 200	0.00643	28 500	0.00667	27 500	0.00560	26 900	0.00548

The modulus of elasticity was determined by drawing a straight line through the mean of the plotted points, below a unit stress of 10,000 to 15,000 lb. per sq. in., of the composite stress-strain curve for each specimen and these values are tabulated in Table I. The modulus of elasticity was not affected by any variation in type of specimen.

Eccentric Loads.—The load was applied to some of the specimens with the spherical-headed loading pins wedged so as to bear on only one side of the spherical seats. This arrangement applied the load at a distance from the longitudinal axis of the specimen, thereby introducing a definite, but unknown, bending moment in the specimen. In practically all cases where the specimen was so loaded, the first few strain-gage readings showed a pronounced difference in the strains on different gage lines, but an adjustment soon began to take place in the loading device and before the stress had become high the increases in the deformation along the different gage lines were equal for a given increment of load. The stress-strain curves for each gage line of specimen 1ZT1SO are shown in Fig. 6. The release of the initial eccentricity is clearly shown by the marked change in the curvature of the lower portion of the curves.

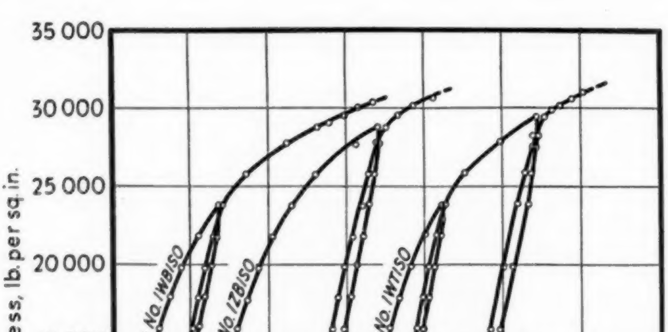

FIG. 7.—Stress-Strain Curves Showing Hysteresis Loops.

The release of eccentricity seems to be caused by a slight bending of the loading pins, by rotation of the screw couplings, and by deformation of the threads in the connections. No arrangement of the spherical bearing block or the loading pins would maintain an eccentric load on the specimen. Most of the specimens loaded eccentrically failed at a slightly lower ultimate strength and unit strain than similar ones loaded axially. It is not certain that eccentric loading was responsible for the lower strengths since the maximum tensile unit strain on any gage line was usually less than that found at failure of similar specimens under axial loads. The effect on strength of eccentricity maintained to rupture needs further study.

Effect of Release of Load.—The load was removed from a number of specimens before failure and strain readings taken at about the same increments in removing the load as were used in applying it. The load was then reapplied in approximately the same increments and strain readings again taken. The release of load was made from different stresses and in one case from about 75 per cent of the ultimate strength and again at about 93 per cent of the ultimate strength. Typical composite stress-strain graphs for each of these cases are shown in Fig. 7. It was noted that the higher the stress from which the release was made, the broader the loop. The significance of these loops is uncertain, though they may suggest that an occasional overload on cast iron does not change the general shape of the stress-strain curve and does not affect the ultimate strength. The loops are quite uniform for the different specimens at the same stress, thus indicating uniformity of action of material and specimen.

Permanent Deformation.—Seven of the forty specimens broke outside the gage length and their permanent deformations were measured by means of strain-gage readings taken after rupture. It was found that the deformation remaining after rupture varied from 38 to 52 per cent of the total deformation at failure. From these data it seems clear that 40 to 50 per cent of the total deformation is permanent as the ultimate strength is approached and will not be recovered if the load is removed.

CONCLUSIONS

From the data presented in this paper the following conclusions appear to be justified with respect to tensile tests of cast iron:

1. Different castings made from the same pouring had a variation of 10 to 11 per cent. The material throughout the length of each casting was fairly uniform, the bottom being stronger than the top by a maximum of 7 to 8 per cent.

2. Hollow and solid specimens had nearly the same strength, with the solid 2 to 5 per cent stronger. In each of these types there was little difference between the specimens with the threads on the outside and those on the inside.

3. The modulus of elasticity was not affected by any method of testing used, but the higher strength casting gave higher values.

4. A solid, machined, specimen with threaded ends tested with spherical bearing blocks which allow an adjustment of the load will give actual strength values unaffected by eccentricity. This type of specimen was found to be the most satisfactory test specimen.

5. Loading and unloading specimens had no pronounced effect on the ultimate strength or the maximum unit deformation of the material.

6. Where the material is loaded nearly to failure and then unloaded there will be a permanent deformation of 40 to 50 per cent of the maximum deformation.

7. Fractures were not localized at any particular section of the specimens but there is evidence that it would have been localized at the change in cross-section if the radius of the fillet had been less than it was.

8. Surface finish is not particularly important in tension. Small superficial defects do not produce stress concentrations which greatly affect strength; the greatest source of stress concentration appears to be caused by an abrupt change in cross-section near the ends of the specimen. A slight increase in cross-section over a short length does not cause a detectable stress concentration.

DISCUSSION

Mr. J. T. MacKenzie.[1]—I consider the work reported in this paper a fine step forward in our knowledge of the tensile strength of cast iron. A few words of warning, however, may be in order. The term cast iron really covers a very wide range. Some of the things that we talk about in soft cast iron will not hold for the harder grades. For example, the summation of the tests for the last 10 yr. that was given in the symposium[2] showed that the relation of the modulus of rupture to the tensile strength dropped from about 2.2 at 20,000 to 1.2 at 55,000 lb. per sq. in. tensile strength, so that with a 50,000 lb. per sq. in. tensile strength iron you might find that eccentricities would be very much more important than with a 30,000 lb. per sq. in. tensile strength iron. I also think that this big specimen might obscure some of the things a person might find in testing. I have determined tensile strengths in certain cast irons where a $\frac{1}{4}$-in. hole drilled down the center of an 0.8-in. bar did not reduce the breaking load at all, so you have to consider the quality of the iron and the relative structure of the periphery and the center. If this specimen is cut from a quarter, you would take the average of all the metal out of the center by drilling a hole down the center, which would not be true of an ordinary cast bar, where you would remove the weakest portion of it by machining out the center; that does not change any of the actual conclusions, but if someone making an ordinary test bar were to think that he could drill out the middle of the bar without increasing the strength, he would be very much mistaken.

In regard to the 40 or 50 per cent permanent set, it is especially true here that you have to take into consideration the quality of the iron, because on the high strength irons, running 60,000 lb. per sq. in., I doubt if it would be a third of that; in fact, in the transverse tests, only 50 per cent were obtained with a Ni-resist alloy which was, you might say, really a ductile material as compared to ordinary cast iron.

I am quite interested to note that the tensile "set" is so much higher than that in transverse bending.

Mr. W. J. Schlick[3] (*presented in written form*).—The study on determinations of the tensile strengths of cast iron as it is presented in the paper, appears to have been divided into two parts.

In the first part 36 round-hexagonal specimens were utilized, tested with flat and V-notch grips "as cast," as cast but with artificial surface defects, and with the central 6-in. length machined down to an unstated diameter. In spite of rather extensive descriptions of this portion of the study, the results presented consist of only the average strengths of the three groups and the conclusion that minor surface irregularities, or surface finish, are not particularly important in tension tests. The varia-

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[1] Metallurgist, American Cast Iron Pipe Co., Birmingham, Ala.

[2] Symposium on Cast Iron, sponsored jointly by the Am. Foundrymen's Assn. and A.S.T.M., *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 115 (1933); also available as separate publication.

[3] Civil Engineer, Engineering Experiment Station, Iowa State College, Ames, Iowa.

tions from the average of 8, 11 and 15 per cent, respectively, in tensile strength would indicate more uniform gripping conditions with both flat and V-notch grips than might occur in many series using hexagonal specimens and grips of these types. In fact, these variations would seem to indicate a close correlation, either studied or accidental, between the angles of the V-notches in the grips and those of the specimens.

Even though "skin effect" was not definitely a part of the study, it would be interesting to know how much the diameters of the machined specimens were less than the "as cast" diameters.

The second phase of the study utilized 40 machined specimens, cut from three castings. These specimens gave a very gratifying uniformity in strength and elastic properties. The conclusion reached by Coker and Filon[4] that the effect of the collars on the "Dalby" type test specimens is negligible supports the authors' conclusion that the collars and gage holes had no material effect upon strength. The uniformity in the stress-strain data for the 6 gage lines as shown by Fig. 3 of the paper attests the care with which the data were taken and the suitability of the threaded-end type of specimen. These strain data were taken on an 8-in. gage length. It seems that the strain at the failure must have been greater than that at other portions of the gage length; consequently it would be interesting to know (if it can be determined) the relationship between the average strain over this gage length and the actual strain at the failure.

It is to be hoped that the "larger study of the mechanical properties of cast iron" will include the problem of the position which the test specimen must have in the cross-section of a larger casting if the result for the test specimen is to represent the average or effective strength of the casting.

MR. WALLACE M. LANSFORD[5] (*by letter*).—The tensile strength of cast iron is difficult to obtain not only because of the careful testing procedure necessary but also because of the effect of the size of the test specimen[6] and the method used in making the test specimen whether cast or machined. For the sake of clarity and brevity, the accompanying Table I showing a summary of the authors' test data is given.

TABLE I.—SUMMARY OF DATA ON TENSION TESTS OF CAST IRON.

Number of Specimens Tested	Shape and Condition of Specimen	Kind of Grips Used	Tensile Strength, lb. per sq. in.	Maximum Deviation from Mean, per cent
	Round-Hexagonal Bars—Cast 12 in. Long			
12	As cast Smooth as could be found	Flat or V notch	23 600	8.0
12	As cast With artificial surface defects 8 to 10 holes 0.05 in. in diameter, 0.05 to 0.10 in. deep at various places on the two circular portions	Flat or V notch	24 700	11.0
12	Middle portion turned down over a length of 6 in., diameter not given	Flat or V notch	22 200	15.0
	Machined from 3 by 3 by 56-in. Castings			
16	Round-solid diameter 0.80 machined	Outside threads	29 500	6.1
8	Round-solid diameter 0.80 in. machined	Inside threads	28 200	2.8
8	Round-hollow outside diameter 0.80 in., inside diameter 0.56 in. machined	Outside threads	27 600	4.3
8	Round-hollow outside diameter 0.80 in., inside diameter 0.56 in. machined	Inside threads	26 900	7.1

As shown in Table I the data given should be divided into two distinct groups since the round-hexagonal bars

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[4] E. G. Coker and L. N. G. Filon, "Photo Elasticity," Cambridge (1931).

[5] Assistant Professor of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[6] Symposium on Cast Iron, *loc. cit.*

were not made of the same materials as the cut or machined specimens. For the same reason, the data from the round-hexagonal specimens which were machined over the middle portion cannot be correlated with the data from the solid round bars tested with threaded ends. It would have been interesting if this comparison could have been made, especially so since cast iron tension test specimens are frequently tested using threaded grips. The tests of the round-hexagonal specimens not only showed the effect of the "skin" but also showed a greater variation from the average when the skin was removed. It would have been interesting to know the relative hardness of the two surfaces and how much the diameter of the original bar was reduced. The data of the second group of specimens, all of which were machined, showed unusually small maximum variations from the average. The uniformity of the strain readings shown in Fig. 6 of the paper, taken along six gage lines when initial eccentricity was placed on the specimen, proves the excellent suitability of the threaded specimen for determining the tensile strength of cast iron. The authors should be complimented for showing so conclusively the remoteness of having any eccentricity in the specimen when using threaded ends and their method of testing.

During the past 10 yr. the writer has had occasion to make many physical tests on cast iron, some of which were tension tests. Two types of tension specimens were used—the threaded end specimen, and a pattern having a reduced mid-section through the ends of which a hole was drilled and the load applied by a hardened steel pin. It was found to be much more difficult to drill the holes perpendicular to and on the long axis of the bar than it was to cut threads on a bar concentric with its long axis. Considerable care must be taken in drilling the holes in the ends of the specimen since eccentricity produced by bad drilling cannot be compensated for. Cutting the threads on a specimen and reducing its diameter are similar operations in machining and make it easy to turn the threads concentric with the axis of the specimen.

It is very gratifying to see such a careful analysis of the use of threaded end test specimens of cast iron presented.

MESSRS. J. O. DRAFFIN[7] AND W. L. COLLINS[7] (*authors' closure, by letter*).—With reference to Mr. MacKenzie's comments, it must be said that all of the conclusions reached in the paper cannot be interpreted to apply to all kinds of cast iron. The term cast iron covers a wide range of material and it can be expected that the results of tests of high-strength iron, a portion of the complete investigation not yet undertaken, might not be in accordance with all the conclusions based on the tests so far made.

A number of the questions raised by Mr. Schlick and Mr. Lansford are similar. The round-hexagonal bars that were machined had their diameters reduced from about 0.71 in. to an average of 0.651 in. Just enough material was removed to insure smooth surfaces. It is doubtful whether the tests show the effect of removing the "skin" because the stress concentrations at the fillets (all but one of the specimens broke there) quite probably prevented the actual determination of the ultimate strength of the material. If the maximum percentage variations from the average tensile strength seem small, they are due to chance or to great uniformity of material.

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[7] Professor, and Instructor, respectively, Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

Mr. Schlick also brought up the question of the amount of deformation at failure that takes place in the immediate vicinity of the fracture. No attempt was made to make such measurements. To do so would be very difficult as it would be necessary to know the location of the fracture before it takes place. However, from a comparison of the measured and extrapolated average unit-strains at failure for the specimens breaking inside and outside the gage length, it is concluded that the localized elongation near the fracture is only a small part of the total elongation of the 8-in. gage length.

A FUNDAMENTAL STUDY OF THE DESIGN OF IMPACT TEST SPECIMENS[1]

By H. C. Mann[2]

Synopsis

Basic data are presented regarding the effect of notch form, velocity, and temperature, on the energy values obtained from the tension-impact test. Material embrittlement at low temperature is shown to be the result of two factors, velocity and form of notch. The deficiencies of the flexural type of specimen are brought out and the necessity for consideration of the velocity effect to reveal the true dynamic properties of a material is emphasized.

Introduction

The subject of impact testing has become one of increasing importance to the engineer and metallurgist, due to the rapid advances in engineering construction which have necessitated the use and development of materials capable of withstanding extreme service conditions involving high rates of force application or dynamic loading.

The impact test apparently is capable of revealing certain differences in material condition which cannot be brought out from any other test, but the test results are seldom found to bear any direct relationship with the performance which might be expected from the part in service.

In practically all investigations, the flexure type of specimen has been used and the test conducted at a single velocity. The form of notch has differed widely, the particular shape and dimensions, arbitrarily selected by each investigator, being those considered to yield results indicative of a certain degree of sensitivity to slight differences in material condition. Without basic knowledge of the fundamental reasons for the wide differences obtained from such tests, it is not at all surprising that the many different conclusions drawn have not been generally accepted.

The principal reasons for this condition are the form of specimen used, and the test procedure. It has always been considered essential to prepare the specimen with a rather sharp notch in the belief that the high stress concentration induced would yield results sensitive to slight variations in material condition. Practically no consideration has been given to the effect of volume, test velocity, or to the fact that the impact test deals with energy changes and not stress alone.

From the fact that the value obtained from the impact test is dependent upon the volume of material participating in the absorption of energy, and this volume is determined by the length of uniform least section, it will be evident that modifying the shape at the base of the notch, either intentionally or from

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[1] Publication approved by the Chief of Ordnance, U. S. Army.

[2] Senior Materials Engineer, Watertown Arsenal, Watertown, Mass.

errors in machining, will therefore vary the volume and also the energy required for rupture.

In the case of the flexure type of specimen, the errors of machining are inherently large, and it is also a practical impossibility to predetermine the volume factor. The resultant energy values, therefore, are largely a measure of the effect of surface and local structural conditions rather than of the true dynamic properties of the material.

TABLE I.—IMPACT TEST RESULTS, FT-LB.

All results represent single tests except where noted.

Notch Length, in.	0.252-in. Diameter				0.357-in. Diameter		0.437-in. Diameter			
	S.A.E. No. 1035 Steel		S.A.E. No. 2340 Steel		S.A.E. No. 1035 Steel		S.A.E. No. 1035 Steel		S.A.E. No. 2340 Steel	
	Actual	Theoretical	Actual	Theoretical	Actual	Theoretical	Actual	Theoretical	Actual	Theoretical
0.025	28.0	30.0	41.1[a]	42.0	59.8	60.0	89.7	90.0	125.0	126.0
0.050	23.7	25.0	35.3[a]	37.0	50.3	50.0	79.5	75.0	113.3[a]	111.0
0.075	19.5[a]	22.0	....	....	45.7	44.0	69.5	66.0	....	....
0.100	19.5	19.0	28.5[a]	30.6	41.1[a]	38.0	64.5	57.0	92.6[a]	92.0
0.125	19.5	19.0	....	....	41.1	38.0	....	....	....	....
0.150	23.7[a]	22.0	....	....	45.7	44.0	64.5	66.0	....	....
0.200	28.0	28.0	36.6	37.0	59.7	56.0	84.7	87.0	111.5	111.0
0.400	59.8	58.0	74.4	74.0	117.0	116.0	176.6	174.0	223.4	222.0
0.600	84.7	87.0	111.5	111.0	176.6	174.0	257.0	261.0	330.5	333.0
0.800	117.0	116.0	....	....	229.1	232.0	346.0	348.0	....	....
1.000	140.0[b]	145.0	125.0[b]	185.0	285.7	290.0	418.0	435.0	459.6[b]	555.0

[a] Average of three tests.
[b] Average of two tests.

TABLE II.—ENERGY PER UNIT VOLUME, FT-LB.

Notch Length, in.	S.A.E. No. 1035 Steel				S.A.E. No. 2340 Steel		
	Actual			Theoretical	Actual		Theoretical
	0.252-in. Diameter	0.357-in. Diameter	0.437-in. Diameter		0.252-in. Diameter	0.437-in. Diameter	
0.025	22 400	23 920	23 900	24 000	32 900	33 400	33 600
0.050	9 480	10 060	10 600	10 000	14 100	15 100	14 800
0.075	5 200	6 100	6 160	5 800	....	....	....
0.100	3 900	4 110	4 300	4 000	5 700	6 200	6 120
0.125	3 120	3 280	....	3 200	....	....	....
0.150	3 160	3 050	2 870	2 930	....	....	....
0.200	2 800	2 990	2 820	2 900	3 660	3 720	3 700
0.400	2 990	2 925	2 940	2 900	3 720	3 720	3 700
0.600	2 820	2 940	2 850	2 900	3 720	3 670	3 700
0.800	2 925	2 860	2 880	2 900	....	....	....
1.000	2 800	2 857	2 786	2 900	2 500	3 060	3 700

The tension type specimen is not subject to these deficiencies, the volume can be controlled and measured, the errors of machining are inherently small, and the specimen is applicable to both static and dynamic tests.

At the Watertown Arsenal laboratories the flexure type of specimen was discarded several years ago, because of the reasons briefly discussed, and the tension form substituted. The justification of this procedure is evident from the results of investigations presented before the Society in 1935[3] and 1936.[4] The earlier work on the tension type of specimen discredited the use of the truncated V notch in favor of the square shouldered form in which the entire volume participation in energy absorp-

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[3] H. C. Mann, "The Relation Between the Tension Static and Dynamic Tests," *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part II, p. 323 (1935).

[4] H. C. Mann, "High-Velocity Tension-Impact Tests," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 85 (1936).

tion could readily be measured and correlated with the static test results. These investigations, however, covered only specimens having notch lengths of 0.1 and 1.0 in. It was, therefore, considered desirable to investigate the effect of intermediate and still shorter notches.

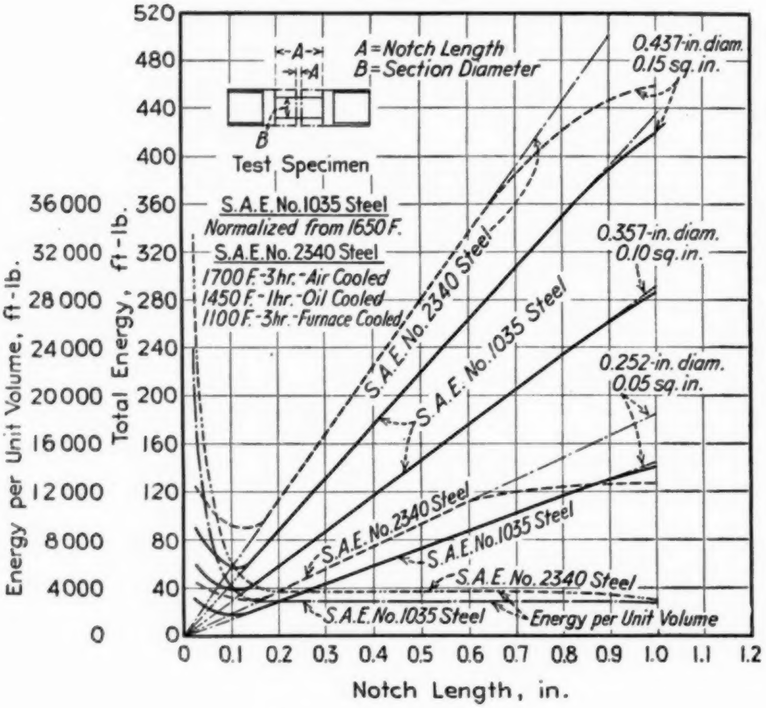

FIG. 1.—Comparison Curves.
Energy-volume relationship tests at 28.5 ft. per sec.

MATERIAL AND TESTS

For this purpose S.A.E. No. 1035 and No. 2340 steels were selected, and, after suitable heat treatment, tension test specimens were prepared having notch lengths varying from 0.025 to 1.0 in., and reduced section diameters of 0.252, 0.357, and 0.437 in., which for each notch length gave volume ratios in the order of 1, 2 and 3. Tests were made in the large 300 kg-m. Charpy impact machine at a velocity of 28.5 ft. per sec., which velocity is less than the transition velocity as determined for the materials in the condition used. The results obtained are tabulated in Tables I and II and plotted in Fig. 1.

The data indicate the existence, within certain limits, of a linear relationship between total energy and notch length, and for any given notch length a relationship between total energy and volume. Theoretical values based on smooth curves drawn through the actual points were determined, and are listed with the test values in the tables. Comparing these data it will be noted that the theoretical values are well within allowable experimental error of the actual test results. It will also be noted that for any given notch length, the total energy is a direct function of the volume. The plotted curves, particularly those indicating energy per unit volume, reveal the presence of three distinct zones: for notch lengths of less than approximately 0.15 in., a zone of energy concentration, increasing to extremely high values as the notch length is decreased; for notch lengths between approximately 0.15 in. and 0.60 to 0.80 in., a zone of linear relationship between energy and volume; and for longer notch lengths a zone of gradual decrease in energy values.

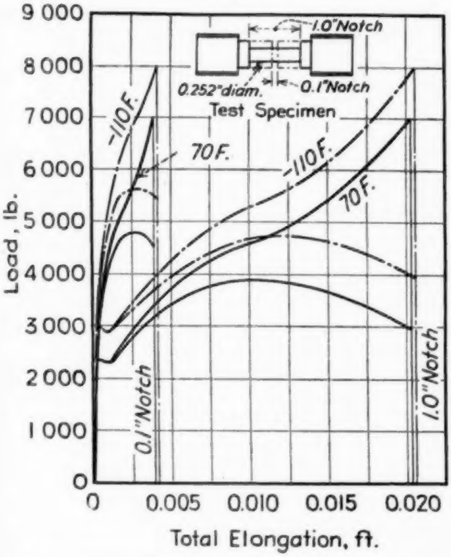

FIG. 2.—Normal and Low-Temperature Tension Static Diagrams.

Results from specimens within the zone of high energy concentration are influenced by slight errors and irregularities of machining, also by the presence of local segregation and nonhomogeneity of material structure, and therefore may not always be indicative of the true dynamic properties of a material. Specimens within and above the zone of linear energy-volume relationship are but little affected by such local conditions, and are in general considered to be more representative of the true dynamic characteristics.

The energy per unit volume curves show that specimens having very short notch lengths are capable of absorbing relatively large amounts of energy when tested in impact under normal temperature conditions. When subjected to low temperatures, however, similar specimens exhibit an embrittling effect which, from previous variable velocity tests, indicates the possibility that low temperature might be a factor influencing such results by virtue of lowering the normal transition velocity of the material.

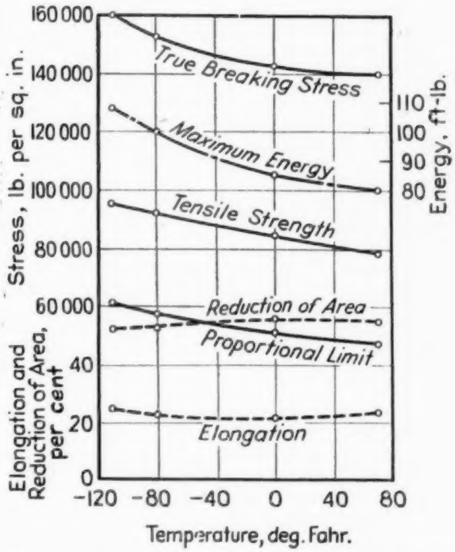

FIG. 3.—Low-Temperature Physical Property Chart.

S.A.E. No. 1035 steel—normalized from 1650 F.; specimens 0.252 in. in diameter, 1.0-in. notch.

To investigate this possibility, specimens of the S.A.E. No. 1035 steel were prepared having 0.252 in. diameter of section and notch lengths of 0.032, 0.10 and 1.00 in. To determine the material characteristics and also the basic or maximum energy values, static tests were first carried out at tempera-

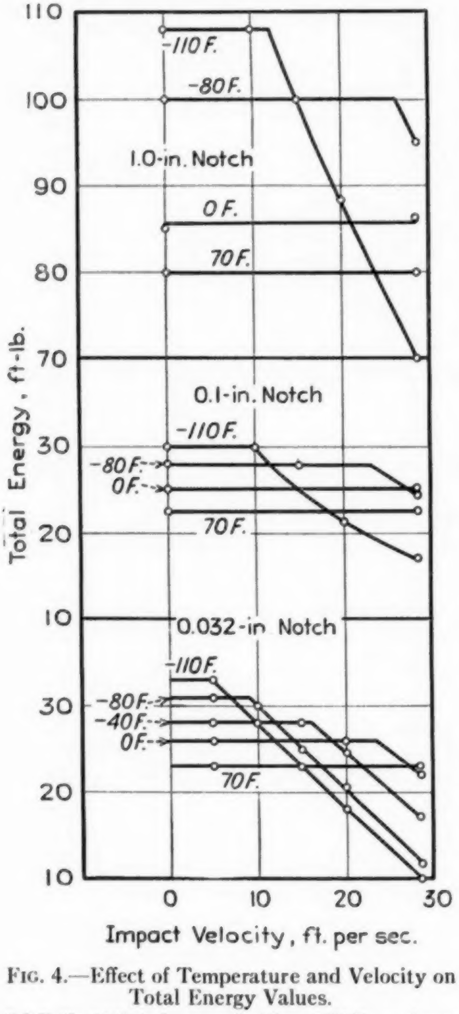

FIG. 4.—Effect of Temperature and Velocity on Total Energy Values.

S.A.E. No. 1035 steel—normalized from 1650 F.; specimens 0.252 in. in diameter.

tures of from 70 to −110 F. These tests were made in a horizontal testing machine, using a special insulated box surrounding the test specimen, to which were attached long holders passing through gland packings at the ends of the box and attached to the heads of the testing machine. By filling the box with acetone to a level well above the specimen and adding dry ice, any desired subtemperature to −110F. could be obtained and held during the entire procedure of rupturing the specimen. The elongation and reduction of area at each increment of load were accurately measured and recorded, from which true-stress or potential energy diagrams were constructed. The diagrams obtained from the 1.0 and 0.1-in. notch specimens tested at 70 F. and −110 F. are shown in Fig. 2. The complete static physical property chart is shown in Fig. 3. The significant feature of this chart is that under static or zero velocity test conditions, there is no evidence of any so-called embrittling effect at low temperatures. The strength values, and maximum energy values calculated from the area under the potential energy diagram, increase with but slight change in ductility, indicating that any lowering of energy values obtained from the impact test must be due to velocity.

In the case of the 0.032-in. notch specimens it was found practically impossible to obtain sufficiently accurate measurements from which to plot a static curve. It was therefore considered that the values obtained from impact tests at 5 ft. per sec. would be sufficiently close to the maximum or static values that they could be used as a basis for comparison.

The impact tests were carried out in the 300 kg-m. Charpy impact machine, a special box surrounding the specimens which were brought down to the desired temperature in a manner similar to the static tests. By this method the specimens were tested while still immersed in the low-temperature bath.

Tests were first made at a velocity of 28.5 ft. per sec. and at the subtemperatures where the energy values obtained were less than the static and 5 ft. per sec. basic values, the impact velocity was successively decreased by lowering the height of fall of the pendulum, until a velocity was reached at which the energy values obtained equaled the basic values. These results are plotted in Fig. 4, and tabulated in Table III.

TABLE III.—LOW TEMPERTURE STATIC AND DYNAMIC PROPERTIES.

Temperature, deg. Fahr.	Test Velocity, ft. per sec.	Proportional Limit, lb. per sq. in.	Tensile Strength, lb. per sq. in.	Elongation, per cent	Reduction of Area, per cent	True Breaking Stress, lb. per sq. in.	Energy, ft-lb.	
							Total	Per Unit Volume
1.0-IN. NOTCH LENGTH								
+70.....	Static	47 000	78 000	23.2	54.5	140 000	80	1 600
	28.5			22.5	54.0		80	1 600
0.....	Static	51 000	84 000	21.8	55.2	143 000	85.2	1 704
	28.5			21.8	55.2		86.7	1 734
−80.....	Static	57 000	92 000	22.7	52.4	153 000	100	2 000
	28.5			22.5	54.0		95^a	1 900
	15.0			21.5	51.9		100	2 000
−110.....	Static	61 000	95 000	24.1	52.0	160 000	108	2 160
	28.5			18.6	52.4		70^a	1 400
	20.0			19.6	51.4		87	1 740
	15.0			22.0	52.4		100^a	2 000
	10.0			23.5	52.0		108.4	2 170
0.1-IN. NOTCH LENGTH								
+70.....	Static	50 000	90 000	19.0	28.0	140 000	22.6	4 520
	28.5			17.0	25.4		22.7	4 540
0.....	Static	54 000	98 000	19.0	23.0	143 000	25.0	5 000
	28.5			17.0	22.0		24.8	4 960
−80.....	Static	60 000	109 000	20.0	22.6	153 000	28.0	5 600
	28.5			13.0	11.0		24.8^a	4 960
	15.0			20.0	21.2		27.6	5 520
−110.....	Static	64 000	114 000	22.0	25.4	160 000	30.0	6 000
	28.5			6.0	3.4		17.0^a	3 400
	20.0			10.0	11.0		21.4	4 280
	10.0			21.2	23.0		29.8^a	5 960
0.032-IN. NOTCH LENGTH								
+70.....	28.5						23.0	14 370
	5						23.0	14 370
0.....	28.5						22.0	13 740
	20						26.0	16 250
	5						26.0	16 250
−40.....	28.5						17.0^a	10 620
	20						24.5	15 300
	15						28.4	17 800
	5						28.0	17 500
−80.....	28.5						11.4^a	7 130
	20						20.8	13 000
	15						25.3	15 800
	10						30.0	18 750
	5						31.0^a	19 370
−110.....	28.5						10.3^a	6 400
	20						18.0	11 250
	15						23.0	14 400
	10						28.0	17 500
	5						33.0^a	21 250

[a] Average of two tests.
All others single test.

It will be noted that the energy values for both the 1.0 and 0.1-in. notches show a decrease at −80 F. when tested at 28.5 ft. per sec. but are equal to the basic values when the impact velocity is reduced to 20 ft. per sec. At −110 F. the values show a very marked decrease and do not come up to the normal basic values until the velocity has been reduced to approximately 10 ft. per sec. In the case of the 0.032-in. notch, the velocity effect is evident at 0 F. and still more pronounced as the temperature was lowered until at −110 F. the maximum or basic value was not attained until the velocity was reduced to 5 ft. per sec.

To illustrate this velocity effect more clearly, the values, in terms of energy

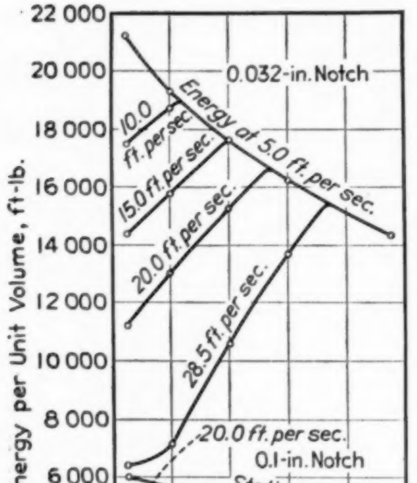

FIG. 5.—Effect of Temperature and Velocity on Energy per Unit Volume Values.

S.A.E. No. 1035 steel—normalized from 1650 F.; specimens 0.252 in. in diameter.

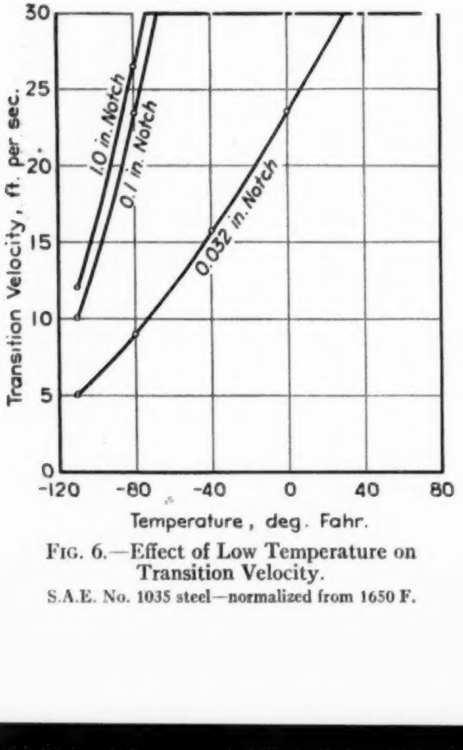

FIG. 6.—Effect of Low Temperature on Transition Velocity.

S.A.E. No. 1035 steel—normalized from 1650 F.

per unit volume, were plotted as shown in Fig. 5. These curves show that within the zone of energy concentration, shortening the notch length and thus increasing the energy concentration, results at any given velocity, in raising the temperature at which departure from the maximum or basic energy value occurs. At the 28.5 ft. per sec. velocity, departure in the case of the 0.032-in. notch occurs at 30 F., while in the 0.1 and 1.0-in. notches, which are just outside the zone of linear energy-volume relationship, the departure is at approximately −70 F. In each case as the velocity was decreased, the point of departure from the maximum or basic energy curve occurred at successively lower temperatures, indicating that the principal effect of low temperature is to depress the normal transition velocity of the material.

TABLE IV.—SHORT NOTCH IMPACT TEST RESULTS.

K 20 normalized from 1650 F. Notch length, 0.020 in.—diameter, 0.252 in. Velocity, 28.5 ft. per sec.

Temperature, deg. Fahr.	Energy, ft-lb.	
	Total	Per Unit Volume
+70	23.7	23 700
+70	23.7	23 700
+40	20.0	20 000
0	15.7	15 700
−40	12.0	12 000
−80	9.2	9 200
−110	7.6	7 600

To show this effect the data from Fig. 4 were plotted as shown in Fig. 6. From the positions of these curves, and also those in Fig. 5, it appears reasonable to assume that low-temperature tests of specimens having notch lengths within the zone of linear energy-volume relationship would be uniform and expected to reveal the true dynamic properties of the material, but when the notch lengths are shorter or within the zone of energy concentration, the results reflect more the influence of local material or surface conditions.

To determine the trend at low temperatures of still shorter notches than heretofore used, specimens having notch lengths of 0.020 in. were prepared and tested at a velocity of 28.5 ft. per sec. The results are tabulated in Table IV and are plotted (Fig. 7) together with those from the 0.10 and 0.032-in. notches in comparison with results from flexure specimens of the same material, having the keyhole and V-type notches.[5]

It will be noted that results from the 0.020-in. notch specimens possess a similarity to those from the Charpy V notch, and results from the 0.032 and 0.10-in. notches to those from the Charpy keyhole notch. In the keyhole notch the dimensions were such that a greater volume participated in the absorption of energy than in the V type of notch which resulted, as in the tension type of specimens, in a lowering of the critical temperature or point of departure from the maximum or basic energy values.

From the curves in Figs. 6 and 7 it may be concluded that in either the tension or flexure types of specimens when the notch lengths are within the zone of energy concentration, the results from low-temperature impact tests are greatly influenced by both notch length and velocity and should not be considered as truly indicative of the material characteristics.

On the basis of the facts presented, it is evident that the disagreement in results from the low-temperature flexure Charpy tests noted in the appendix to the 1936 report of the Joint Research Committee on Effect of Temperature on the Properties of Metals,[5] are due to differences in the notch lengths of the specimens and velocities of the testing machines used.

DISCUSSION AND CONCLUSIONS

To determine the true dynamic properties of a material, the following requirements as to the type of specimen and method of test should be adhered to:

1. The test specimen should be of a type that can be readily tested under static as well as dynamic conditions, and yield data from which maximum or basic energy values can be determined.

2. The reduced or notched section should be of such form that it can be readily machined without introducing errors or irregularities of sufficient magnitude to affect noticeably the test results, and of such dimensions that the volume participating in the absorption of energy can be definitely controlled and measured.

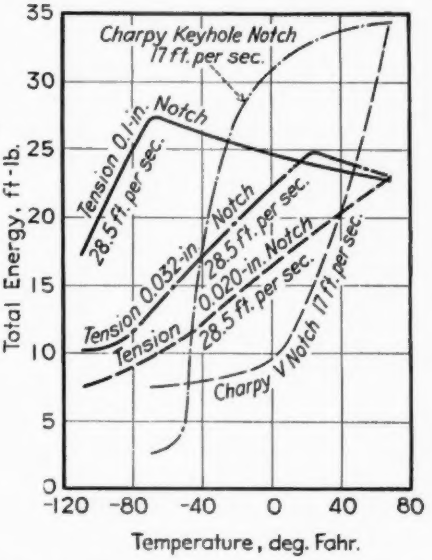

FIG. 7.—Comparison Between Tension and Flexure Low-Temperature Impact Tests.
S.A.E. No. 1035 steel.

3. The length of reduced or notched section should be within the zone of linear relationship between energy and volume.

4. A sufficient number of specimens

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[5] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 132 (1936).

should be tested at different velocities in order that the transition point can be definitely determined.

The tension type of specimen is best suited to meet these requirements. It can be tested under static as well as dynamic conditions, and is capable of furnishing data from which maximum or basic energy values can be determined. It is inherently free from machining difficulties, and such errors of dimensions or irregularities normally produced have no noticeable effect upon the test results; also the volume participating in the absorption of energy can be definitely controlled and measured.

The generally accepted type of flexure specimen is not suited for test under static conditions, since data cannot be obtained for the determination of maximum or basic energy values. The form of notch is difficult to produce and duplicate, and is also inherently subject to errors and irregularities of machining which measurably affect the test results. The volume factor cannot be definitely controlled or measured, and also the length of notch is usually within the zone of energy concentration.

The present types of impact test machines operate at one velocity, which is below the transition point of most materials. Since velocity is one of the most important factors it is essential that the impact test machine be so constructed that high and variable velocities can be obtained.

One of the principal reasons for the continued use of the single velocity impact test appears to be from the fact that certain steels, particularly of the nickel-chromium type, when slowly cooled from the drawing temperature, exhibit a so-called "temper-brittle" condition which, although not evident from the static test, is revealed by the low energy values obtained from the impact test. Variable velocity tension impact tests of such "temper-brittle" material, carried out at the Watertown Arsenal, have conclusively shown that in this condition the transition velocity is extremely low, less than 5 ft. per sec., which accounts for the low impact values obtained from the ordinary single velocity test. Similar conditions have been observed in tests of certain non-ferrous materials and conclusively indicate the necessity for a change in the present methods of impact test.

The phenomenon of the change in transition velocity produced by low temperature as noted in Fig. 6, suggests the probability of a microstructural change in the material from the fact that the true-breaking stress and other physical properties definitely change as the temperature is lowered (Fig. 3). Studies at the Watertown Arsenal, comparing true-breaking stress values with the microstructure of various specimens of different compositions and conditions, have shown a definite correlation. Also, it has been established[6] that the transition velocity is a function of material structure and can be widely varied by heat treatment.

Assuming from these facts that a gradual change in microstructure is produced at lowered temperatures, it would be expected that, at a certain critical temperature dependent upon the material, this change might have progressed to a condition which would affect the transition velocity in the manner noted. In the case of the material tested, this critical temperature is approximately −75 F., as revealed from specimens closely within the zone of linear energy-volume relationship. The influence of energy concentration is to raise this critical temperature, and would

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[6] G. F. Jenks, "Application of Impact Tests," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 59, May, 1937, pp. 313–318.

therefore appear to account for the fact that results from impact tests of specimens having notch lengths within the zone of energy concentration are usually variable, and not indicative of material serviceability under similar low-temperature conditions.

It appears evident from the facts presented that, as a means for determining the true dynamic properties of a material, the present generally accepted methods of impact test are fundamentally incorrect, and have greatly retarded advancement to a thorough understanding of the subject, however establishing the influence of the basic factors, velocity, notch length, and volume, offers a possibility for the early development of an acceptable standardization of the impact test.

Acknowledgment:

This study of the fundamental dynamic properties of materials was made possible from the active interest and cooperation of Col. G. F. Jenks, Ordnance Department, U. S. Army, former Commanding Officer, Watertown Arsenal.

DISCUSSION

Mr. Jesse J. Shuman.[1]—There is a group in the Association of American Steel Manufacturers, a committee on physical tests, which during the past year or two has done considerable investigational work on this problem. It is desired by some of the oil interests that tubular materials used in oil-well production should have high impact values as an additional guide to the life expectancy of the material; but it happens that the standard test specimen, that is, the transverse impact specimen, is 10 mm. square, which of course is thicker than the walls of most tubes. The problem therefore has been to evaluate reduced size impact specimens. The literature on that subject has indicated that results on sub-size specimens do not follow the arithmetical rule; that is, a specimen one-half of the standard thickness does not give results half as great as the standard specimen, but it may be 60 to 70 per cent instead of 50 per cent of the expected results. The committee wished to determine whether a tension impact test which could of course have its specimen from the wall of any tube, might not answer the purpose and give the same indications. After going through an extensive experiment in which some nine laboratories exchanged specimens and results, it was rather apparent that the tension impact specimen as made on the ordinary Charpy pendulum machine is not a suitable substitute for the transverse impact specimen as made with the keyhole notch. We were hoping in that committee that the extensive work at Watertown Arsenal with tension impact tests might point out the next step that could be taken in order to provide a suitable procedure of impact testing which will be dependable in indicating the impact values of these important steels. The tonnages involved are large and the matter continues to be one of importance, and if Mr. Mann or his associates can guide us in the direction that our investigational work should go, it would be greatly appreciated. The specimen that seemed to the committee to be best adapted for testing pipe in tension was one having a $\frac{1}{4}$-in. diameter over a parallel turned length of 1 in., measuring the elongation in $\frac{3}{4}$ in. It was necessary to decide on some standard, and that happened to be the standard that was worked out.

Mr. G. F. Jenks.[2]—In answer to the last speaker, the Ordnance Dept. would be very glad to examine the test data obtained on pipe and give its opinion as to whether some modified form of specimen or modified method of tension impact test might not be more applicable. I am inclined to believe that the specimen used was not properly designed; it would probably have been necessary, in order to obtain high stress concentration, to have used a shorter notch.

Mr. D. L. Smart.[3]—The subject of impact testing is of particular interest to The Detroit Edison Co. because of

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[1] Inspecting Engineer, Jones & Laughlin Steel Corp., Pittsburgh, Pa.

[2] Colonel, Ordnance Dept., U. S. Army; Chief of Technical Staff, Office of the Chief of Ordnance, Washington, D. C.

[3] Research Dept., The Detroit Edison Co., Detroit, Mich.

its interest in the behavior of ferrous metals subjected to high-temperature service. We have operated experimental steam superheaters at 1000 and 1100 F. for as long as 40,000 hr., removing samples of metal from time to time in order to determine the changes in physical properties and microstructure. One of the physical properties with which we were most concerned was impact strength.

In interpreting the results of impact tests, however, we have frequently been faced with the same difficulty mentioned by Mr. Shuman in that it has not always been possible to use standard 0.394 by 0.394 in. V-notch Izod specimens. When the source of samples was $\frac{3}{8}$-in. wall thickness tubing, we have reduced the width across the notch to 0.340 in., keeping all other dimensions the same, and when the source was $\frac{1}{4}$-in. wall thickness superheater tubes, we have had to reduce the width still further to 0.215 in. We were forced to attempt to draw conclusions regarding the effect of different lengths of service from the results of tests using two different non-standard sizes of impact specimens and perhaps to try to correlate our results with those of another investigator who used standard specimens. This was not satisfactorily done.

We made a study, therefore, of the impact strength of a 0.30 per cent carbon, silicon-killed steel when using the three types of specimens already referred to and a fourth of still smaller size. We found that as we reduced the specimen width, keeping all other dimensions the same, the impact strength increased considerably. Tests of 18 per cent chromium, 8 per cent nickel steel, however, have indicated a decrease in impact strength in almost direct proportion to the decrease in specimen width, and tests of carbon-molybdenum steel have shown only a slight increase in impact strength for the narrower specimens. It is apparent that if a factor is found by which the impact strength of a given steel, determined from non-standard specimens, could be corrected for specimen size it would not apply to all other steels.

In view of all this, we have hoped that the tension impact test might furnish a means by which we could make impact tests on specimens of different sizes and compare the results in a way in which we are not able to do at present. Some of the results presented by Mr. Mann showed that, for specimens of a given diameter, the impact energy per unit of volume remained practically constant for notch lengths above a certain minimum. What we are anxious to ascertain is whether we can use different diameters and also different notch lengths and obtain constant energy absorption per unit of volume.

MR. H. J. GOUGH.[4]—While I have not yet studied Mr. Mann's paper, I have seen something of this work at Watertown Arsenal. I think this entry into the field of tension impact testing is of great importance in the history of impact testing. On the other hand, I certainly do not agree with Mr. Mann's concluding remarks that his work has shown the method of the flexural notched-bar impact test to be fundamentally wrong. In England we do not profess yet to understand the notched-bar test but a great amount of experience has established its value as a discriminating test for heat treatment, cold work, etc. If the author had exposed the shortcomings of the notched-bar test or established his own results on a rational basis, there might be some justification for his concluding remark; I cannot see that he has done either. As I under-

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[4] Superintendent, Engineering Dept., National Physical Laboratory, Teddington, Middlesex, England.

stand the position, the energy absorbed in a static tension test—as determined by the integration of the load-elongation diagram—*cannot* be correlated with the energy absorbed to fracture in tension impact.

To meet that difficulty, the author alters the static diagram by correcting for the reduction in area of the specimen, integrates the resulting curve, and states that the area under this curve now agrees with the energy absorbed in the impact test. But the value obtained from the corrected curve has no real meaning; it cannot express the total energy absorbed by the specimen to fracture. Hence the correspondence claimed must be regarded merely as a coincidence requiring explanation; there is no justification for the statement that the energies absorbed in both types of test have identical values. Until this difficulty has been overcome, the results given must merely be accepted as interesting experimental data not yet capable of interpretation. I would suggest that until this rationalization has been achieved it would be unwise and unwarranted to dismiss the enormous amount of experience already obtained with the flexural notched-bar test.

That is not to imply that the position of knowledge of notched-bar testing is regarded as satisfactory; to cite merely one example, a serious attempt to investigate the test on a dimensional basis was a complete failure. But its value as a simple and quick test to distinguish differences in microstructure and degree of cold work has been established for a great variety of steels; also, abnormal impact values have often been found associated with failures in service. So we shall watch the investigation of the tension impact test with very great interest, sympathy, and admiration, but, until it is placed on some real physical basis, we are not prepared to abandon the notched-bar test.

MR. H. C. MANN[5] (*author's closure, by letter*).—From the remarks of Mr. Shuman and Mr. Smart, it was apparently expected that the results of these investigations would suggest a form of tension specimen which, when tested under ordinary impact methods, would yield more dependable results than the flexure type. Although results comparable to the flexure type of specimen can be obtained by selecting the proper form of tension specimen, neither will reveal the true dynamic properties of a material unless the velocity factor is considered.

These investigations have clearly shown that velocity is a most important factor and one which must be considered in determining dynamic characteristics. The velocity effect, or transition point, is, in general, only revealed from tests conducted at velocities higher than those of the ordinary impact machine. Since the single velocity test gives only one point on the energy-velocity curve, it should be evident that the results can be of little value if, in service, the rate of loading or force application is at a velocity other than that at which the test is made.

It is believed that considerable progress could be effected if industry would consider that the fundamental dynamic properties of a material must first be determined from variable velocity and temperature tests, and, having once established these characteristics, it should be possible to set up simple acceptance test limits. The research investigations at Watertown Arsenal are being conducted with this in view, and the results, which correlate transition velocity with carbide condition and

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[5] Senior Materials Engineer, Watertown Arsenal, Watertown, Mass.

magnitude of energy values with ferritic distribution, are most promising and have added greatly to our understanding of the subject.

From the remarks of Mr. Gough, it is quite evident that the method of presenting the theory of the static-dynamic relationship has been such that it is not readily understood unless one has carefully studied in detail the basic data presented. Since acceptance of the factual data on this subject apparently hinges on the static-dynamic relationship, a more detailed explanation of the phenomena is presented.

From a series of static and dynamic tests of a given material and condition using similar specimens, it was found that during deformation the rise in temperature at any section normal to the axis of the specimen was directly proportional to the change in area at that point, and that the heat evolved was the same in both types of test.

In the static test, however, measurements of load and elongation are taken only after equilibrium or zero velocity conditions have been established, that is, after all heat evolved has been dissipated. The test, therefore, is isothermal in nature, and the load-elongation diagram represents simply strain-energy, since the load maintained is dependent only upon the elastic properties of the material. In the dynamic test, rupture takes place without appreciable heat dissipation, and the test, therefore, is adiabatic in nature.

Since under both static and dynamic conditions all measurable properties—elongation, reduction of area, and heat evolved—are the same, it is evident that the factor determining whether the test is isothermal or adiabatic in nature is velocity.

For isothermal (static) conditions, the total energy value may be expressed as:

$$\delta Q = \delta W_S$$

and for adiabatic (dynamic) conditions:

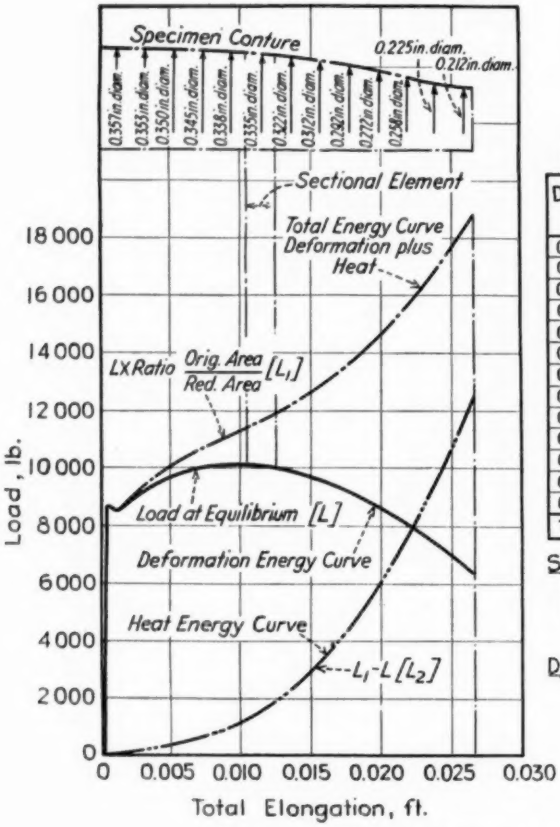
$$\delta Q = \delta W_S + \delta W_H$$

where δW_S = strain-energy, and

δW_H = heat-energy.

In the static test it was observed that to produce a given amount of deformation, if the rate of load application was increased, the momentary observed load and the temperature was higher than at equilibrium. As the heat dissipated, the temperature reduced to normal and at the same time the load dropped off to that required for elastic balance. For relatively small increments of deformation, it was found that the rate of loading could be increased to the point above which no greater rise in temperature or increase in observed load could be obtained. At this point the temperature rise was found to be the same as obtained from the dynamic test, producing an equal amount of deformation, and indicated the attainment of an adiabatic condition.

It is only during the process of deformation that heat is evolved, and the amount of heat produced was found to be directly proportional to the change in sectional area. In the case of the static or isothermal diagram, the increment load values are only those sustained by the elastic strength of the reduced section, and do not represent the additional loads necessary to produce the same increments of deformation under adiabatic conditions. From this it was assumed that the increment total loads under adiabatic conditions could only be those which would be sustained by an area equivalent to the original cross-section, but having the same elastic strength as the reduced section, and a diagram constructed on this basis should represent the true dynamic or adiabatic conditions. This diagram is readily constructed by increasing the static load values in the ratio of original to reduced sectional areas. Results from a large number of tests of ferrous and non-ferrous materials conducted contour shows the increment dimensional changes produced during the progress of deformation to rupture. The length of the sectional element used in the energy calculations was 0.025 in., which included the change in similar elements from both halves of the specimen.



Energy, ft-lb. per Sectional Element

Diam., in.	Calculated From	
	Heat Evolved	Load-Elon. Diagram
0.357		
0.353	0.006	0.08
0.350	0.058	0.20
0.345	0.227	0.32
0.338	0.670	0.67
0.335	0.760	0.78
0.322	2.060	2.00
0.312	3.260	3.30
0.292	6.300	6.32
0.272	10.710	10.69
0.258	13.700	13.70
0.225	20.430	20.50
0.212	25.000	25.00
Total	83.181	83.56

Static

Deformation	243 ft-lb.
Heat	83 ft-lb.
Total	326 ft-lb.
Dynamic	325 ft-lb.

FIG. 1.—Comparison of Energy Values.

under normal, and both high- and low-temperature conditions, have substantiated and verified the correctness of the above assumption.

A typical example, with a comparison of energy values determined from actual temperature measurements and the revaluated diagram, are shown in the accompanying Fig. 1. The specimen

This correlation between static and dynamic tests on the basis of isothermal and adiabatic conditions was originally presented early in 1935.[6] Further tests altering the original concept were the basis of a subsequent paper presented

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[6] H. C. Mann, "Relation of Impact and Tension Tests of Steel," *Metal Progress*, March, 1935, p. 36.

before this Society.[7] A recent complete check of all data, however, brought out an error of calculation which, when corrected, verified the original results which were essentially the same as covered in this discussion.

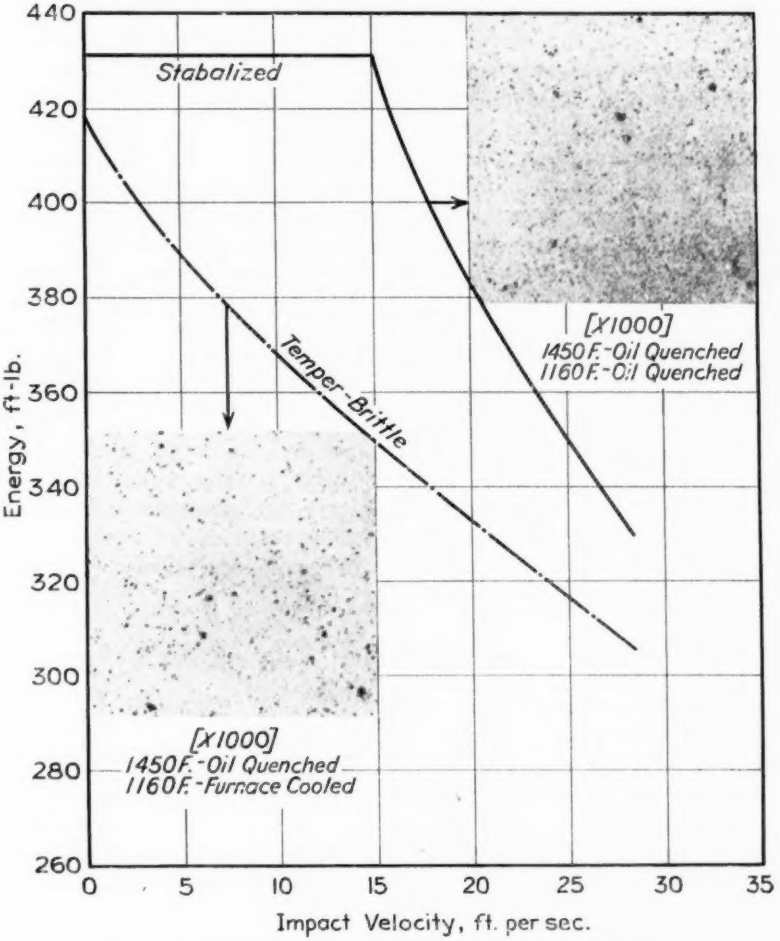

FIG. 2.—Effect of Carbide Condition on Transition Velocity.

S.A.E. No. 3135 steel—stabilized and temper-brittle; specimens 0.357 in. in diameter by 1.4 in.

As to the value of the ordinary flexural notched-bar test, in a résumé[8] of the 1922 Symposium on Impact Testing, it is stated that, "the results are not sufficiently discriminating to indicate any but rather large differences in materials." From the vast amount of impact testing carried out at the Watertown Arsenal Laboratories, nothing has been revealed that would nullify this statement.

Where there is a marked difference between the ordinary impact values of correctly and incorrectly heat-treated

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[7] H. C. Mann, "The Relation Between the Tension Static and Dynamic Tests," *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part II, p. 323 (1935).

[8] Résumé of the Facilities and Methods for Making Impact Tests, and Their Interpretation, *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part I, p. 546 (1926).

material, the variable velocity test has shown that when incorrectly treated, the material has an extremely low transition velocity. This is illustrated in the typical example shown in the accompanying Fig. 2. At the velocity of the ordinary impact machine (16 ft. per sec.), the difference in energy values is clearly evident and the flexural test would naturally reveal this condition. However, many other cases have been found where the material has been heat treated to have a transition velocity of over 100 ft. per sec., but when differently processed a transition point of only 30 ft. per sec., below the transition point the energy values being approximately the same. In such cases the ordinary single velocity impact test would show no difference between the two conditions.

The results of further continuation of the variable velocity impact investigation have revealed the fact that transition velocities are markedly affected by grain boundary carbide conditions; when present as continuous or intermittent chains, the transition point is low, but when well diffused and not at the grain boundaries, it is greatly increased. The photomicrographs in Fig. 2 show this comparison. In all these studies no correlation has been found between transition velocity and usual micro characteristics. The only correlation so far noted has been that the condition of ferritic distribution appears to govern the magnitude of the impact energy value.

In view of the data presented, it would appear unnecessary to discuss further the shortcomings of the ordinary single velocity notched bar test. The obvious general lack of correlation with any known physical property of the material, or with subsequent service behavior, should be sufficient evidence of the unreliability of the test as usually conducted. In support of the proposed method of variable velocity testing, there is the following indisputable fact: one point (a single test at one velocity) does not determine the shape of the energy-velocity (dynamic) curve.

ANALYSIS OF THE BRINELL HARDNESS TEST

By Robert H. Heyer[1]

Synopsis

Certain features of the Meyer hardness test are reviewed.

A method is proposed for expressing a shear yield strength which bears a definite relationship to tensile or compressive yield strengths.

An explanation is offered, based on relative strengths in shear and compression, for the differences commonly observed in the surface contours of Brinell impressions. Other differences in the nature of the plastic deformations below the surface of the impression are studied by means of a split hardness test specimen.

Relations are established between depth of visible plastic deformation, rise of metal above the original surface, and the Meyer **n** coefficient. The rate of increase of depth of plastic deformation with diameter of indentation is determined for four series of metals. The published work on thickness requirements is reviewed from the standpoint of the present determinations.

Data are presented on the effect of annealing on many of the hardness properties of a cold-drawn silicon bronze.

Plastic deformations under cylindrical and spherical indentors are compared.

Introduction

Over a period of 37 yr. following its introduction by J. A. Brinell, there has been a considerable amount of research on the Brinell hardness test and on the correlation of hardness with the results of other less convenient methods of physical testing. A very large proportion of this literature has been summarized in Hugh O'Neill's monumental book on hardness testing.[2]

In view of these facts, it is rather surprising to discover how little is known of the fundamental mechanics of a ball hardness test. Although no claims are made for a fundamental solution at this time, it is hoped that the present work will prove to be a step forward.

The ball type of indentor was chosen in preference to the conical or pyramidal types partly because the Brinell type test is best adapted to the separate evaluation of two factors which enter into any type of indentation test: namely, the pretest hardness and the rate at which hardening takes place during the test.

Meyer[3] discovered a practical method for making such a division in his pioneering investigation reported in 1908. Although the main purpose of the present

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[1] Member, Research Dept., The American Rolling Mill Co., Middletown, Ohio. Based upon a thesis to be submitted by Robert H. Heyer to the Faculty of Purdue University in partial fulfillment of the requirements for the Degree of Doctor of Philosophy, June, 1938.

[2] Hugh O'Neill, "The Hardness of Metals and Its Measurement," The Sherwood Press, Cleveland, Ohio (1934).

[3] E. Meyer, "Untersuchungen über Härteprüfung und Härte," *Zeitschrift* des Vereines deutscher Ingenieure, Vol. 52, pp. 645, 740, 835 (1908).

paper is to describe the nature and significance of the Brinell impression itself, the Meyer analysis is indispensable in discussion of the results; consequently some of its features are summarized here.

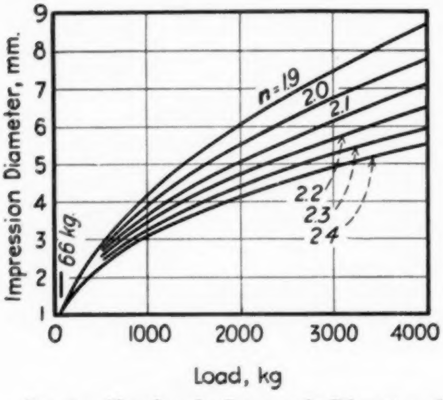

FIG. 1.—Showing the Increase in Diameter of Impression with Applied Load for Six Metals Having Constant "Unworked Hardness" ($\mathbf{a}$ = 66) but Variable Work-Hardening Coefficients.

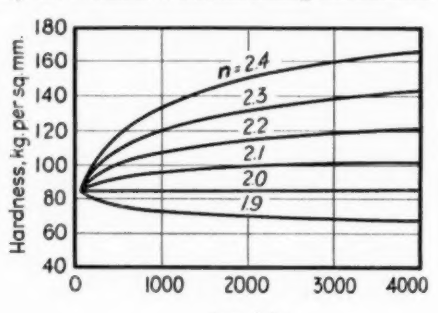

FIG. 2.—Showing Variation in Meyer Hardness (Eq. 2) with Applied Load for the Six Metals of Fig. 1.

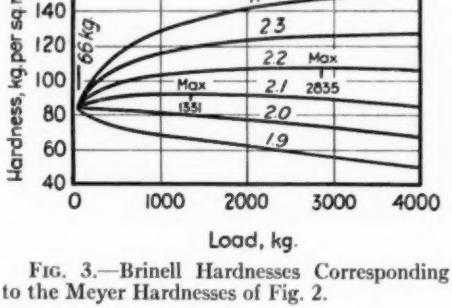

FIG. 3.—Brinell Hardnesses Corresponding to the Meyer Hardnesses of Fig. 2.

The Meyer test method, although universally accepted, has been used relatively little in this country. The relationship expressed in Eq. 1 below is capable of almost universal application to ball-type tests, and very few cases have been reported wherein experimental data have deviated from this exponential relationship, within the limits of impression diameters originally set.

O'Neill[2] and Hoyt[4] have given us excellent accounts of Meyer's work, which may be summarized in the expressions:

$$L = \mathbf{a}d^{\mathbf{n}} \qquad (1)$$

$$M = \frac{L}{\pi d^2/4} \qquad (2)$$

where

L = load in kilograms (usually on a 10-mm. ball),

d = diameter of indentation in millimeters,

M = Meyer hardness in kilograms per square millimeter of projected area,

$\mathbf{a}$ = pretest hardness in kilograms per square millimeter of projected area, and

$\mathbf{n}$ = coefficient representing strain-hardenability.

Figures 1 to 3 are presented as visual aids to interpretation of the constants $\mathbf{a}$ and $\mathbf{n}$, and for direct comparison of M (Meyer hardness) with the more usual Brinell hardness. In these figures the constant $\mathbf{a}$ has been arbitrarily taken to be 66, the value for mild steel in the data following. Thus in each figure six metals are represented having a constant unworked hardness but different rates of strain-hardening, represented by values of the strain-hardening

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[4] S. L. Hoyt, "The Ball Indentation Hardness Test," *Transactions*, Am. Soc. Steel Treating, Vol. 6, p. 396 (1924).

coefficient, **n** ranging from 1.9 to 2.4. The value 66 for constant **a** may be given the following approximate interpretation. When $d = 1$, $L = \mathbf{a}d^{\mathbf{n}} = 66\ (1)^{\mathbf{n}} = 66$ kg. required to produce a 1-mm. diameter impression in each of the six metals, independent of **n** values. Considering the shallow nature of a 1-mm. diameter impression as applied by a 10-mm. ball, and the small amount of work-hardening produced, we are perhaps justified in associating this constant with the pretest hardness. However, in order to appreciate the magnitude of the stresses and deformations when $d = 1$ mm. and $L = \mathbf{a} = 66$ kg., the following calculations from elastic theory were made. Using the development by Thomas and Hoersch,[5] an elaboration of the Hertz theory, the load and *elastic* impression diameter at the point of elastic failure due to internal stresses were calculated from the physical data available for the mild steel being considered. Using 25,800 lb. per sq. in. as the yield strength in shear (defined on page 124), it may be shown that a load of 0.37 lb. on a 10-mm. steel ball will produce yielding. The corresponding elastic impression will be 0.0031 in. in diameter; the maximum compressive stress just under the ball will be 80,200 lb. per sq. in.; and the mean effective pressure (based on the projected area) will be 49,200 lb. per sq. in. Furthermore, the yielding will start at a point 0.00072 in. below the surface, at which point the shear stresses are at a maximum. (Thomas and Hoersch checked similar applications of their formulas by experiments with mild steel, using much larger indentors than considered here.)

The calculated values of maximum and mean effective compressive stress are both considerably greater than the compressive yield strength of the mild steel (found to be 34,700 lb. per sq. in.). In fact, 80,200 lb. sq. in. is greater than the tensile strength for this material, yet permanent yielding is negligible at the beginning of elastic failure.

Returning from elastic behavior to the permanent indentations of the hardness test, the calculated mean effective pressure when $d = 1$ mm. and $L = 66$ kg. is 84 kg. per sq. mm. or 119,500 lb. per sq. in., and the maximum stress just under the ball must be somewhat higher. It is not likely that the strain-hardening effect under a 1-mm. diameter impression is sufficient to raise the strength of even a small portion of the metal to such a figure.

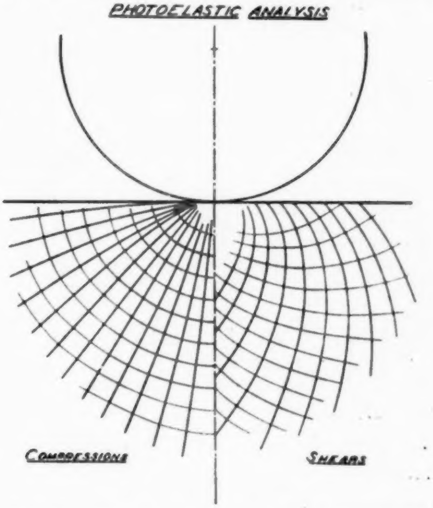

FIG. 4.—Photoelastic Analysis of Stress Directions in a Celluloid Plate Loaded Through a Celluloid Cylinder.

Obviously the directions of the stresses below the surface are not parallel and in the direction of the external load, but radiate from the area of contact so that the effective area supporting the load is considerably greater than the projected area of contact. This has been proved for elastic conditions

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[5] H. R. Thomas and V. A. Hoersch, "Stresses Due to the Pressure of One Elastic Solid upon Another," *Bulletin No. 212*, University of Illinois Engineering Experiment Station (1930).

by Coker[6] and Mesmer. (Reported by Nadai).[7] The results of an additional photoelastic determination of the stress trajectories in a celluloid plate loaded through a cylinder of the same material are given in Fig. 4. The mathematical solution for the case of a single concentrated force acting at the edge of a large plate (an infinite plane) indicates that the trajectories of the primary compressive stresses are straight lines radiating from the point of contact.[6] The curvature of the compression lines in Fig. 4 indicates a finite area of contact of the celluloid cylinder and plate. These stress trajectories may be compared with those obtained under plastic conditions (see Figs. 7 and 8).

It is interesting to note that a load of 0.46 lb. would produce a *permanent* impression 0.0031 in. in diameter, equal in size to the elastic impression at 0.37-lb. load, provided the Meyer relationship holds down to such low loads. This is a debatable assumption. Takahasi[8] presented data showing that **n** approached 2.0 as *d* approached zero. However, Krupkowski[9] made very extensive tests on copper using more sensitive means of loading, a lower minimum load (1 kg.), a longer time of application of the load (3 min. compared with 20 sec.), and diameter rather than depth measurements. Krupkowski concluded that the Meyer exponential formula is exact for copper in the load interval 1 to 10,000 kg., and for balls ranging from 1 to 30.16 mm. in diameter.

The significance of the constant **n** should be quite apparent from a study of Figures 1 to 3.

Figure 1 shows that the impression diameter increases with load at a relatively low rate in the case of a metal with a relatively high work-hardenability; for example, **n** = 2.4.

Figure 2 shows the corresponding calculated values of Meyer hardness for the six metals, in which the metal of highest work-hardenability reaches an apparent hardness nearly twice as great as a metal with no work-hardenability (**n** = 2.0), when both are tested at the usual load of 3000 kg.

Figure 3 shows calculated Brinell hardness numbers for corresponding diameter-load readings. The apparently meaningless maximum values for Brinell hardness have several times before been used as evidence against the scientific accuracy of the empirical Brinell relationship. Severely cold-worked metals having values of **n** approximately = 2.0 show decreased Brinell hardness values with increasing loads in the entire range of loading. Figure 3 also illustrates the fact that in the case of most commercial steels the hardness values will be reasonably constant at various loads because their **n** values usually lie between 2.15 and 2.25 unless they have been drastically cold worked. It is believed that this fact influenced Brinell in his selection of the surface area of the indentation instead of the simpler projected area.

MATERIALS

Several representative, commercially available alloys were obtained in the form of bars $\frac{7}{8}$ or 1 in. in diameter. In addition to the hot-rolled, cold-drawn, or extruded state, the bars were tested after certain heat treatments as indicated in Table I. This group covers a wide variety of types of hardness impressions and Meyer coefficients, the principal deficiency in the list being hardened and tempered machine steels

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[6] E. G. Coker, "Contact Pressures and Stresses of Curved Members," *Proceedings*, Inst. Mechanical Engrs. (British), p. 901 (1928).

[7] A. Nadai, "*Plasticity*," p. 254, McGraw-Hill Book Co., Inc., New York City (1931).

[8] K. Takahasi, "Relation Between Pressure and Diameter of Impression in Brinell Hardness Test," *Metal Stampings*, Vol. 2, p. 888 (1929).

[9] A. Krupkowski, "Mechanical Properties of Copper," *Revue de Metallurgie*, Vol. 28, pp. 529, 598, 641 (1931); Vol. 29, pp. 16, 74 (1932).

having much higher a constants than those found here.

TESTS METHODS

It was considered desirable to determine relative yield strength values in compression and in shear as an aid to interpretation of certain hardness characteristics; therefore compression and torsion tests were carried out according to the following procedures.

TABLE I.—DESCRIPTION OF MATERIALS TESTED.

Series	Designation	Description	Source
No. S1......	Armco A	Armco ingot iron. Hot rolled	The American Rolling Mill Co.
No. S2......	Armco B	Armco ingot iron. Annealed at 1560 F.	The American Rolling Mill Co.
No. S3......	Mild steel A	0.15 per cent carbon. Cold drawn	Stock
No. S4......	Mild steel B	0.15 per cent carbon. Quenched in water from 1660 F. Spheroidized at 1300 F. for 21 hr.	Stock
No. S5......	Mild steel C	0.15 per cent carbon. Quenched in water from 1660 F.	Stock
No. S6......	Mild steel D	0.15 per cent carbon. Annealed at 1660 F.	Stock
No. S7......	S.A.E. No. 2330 A	S.A.E. No. 2330. Hot rolled	Stock
No. S8......	20% Cr A	0.11 per cent carbon, 19.6 per cent chromium stainless steel. Hot rolled	Crucible Steel Company of America
No. S9......	20% Cr B	0.11 per cent carbon, 19.6 per cent chromium stainless steel. Cooled in air from 1500 F.	Crucible Steel Company of America
No. S10.....	18-8 A	0.07 per cent carbon, 17.6 per cent chromium, 8.7 per cent nickel stainless steel. Hot rolled	Crucible Steel Company of America
No. S11.....	18-8 B	0.07 per cent carbon, 17.6 per cent chromium, 8.7 per cent nickel stainless steel. Quenched in water from 2000 F.	Crucible Steel Company of America
No. S12.....	Monel A	Monel metal. Cold drawn	International Nickel Co.
No. S13.....	Monel B	Monel metal. Cooled in air from 1650 F.	International Nickel Co.
No. S14.....	Herculoy A	Silicon bronze. Cold drawn	Revere Copper and Brass, Inc.
No. S15.....	Herculoy B	Silicon bronze. Annealed at 800 C.	Revere Copper and Brass, Inc.
No. S16.....	Brass A	Free cutting, leaded brass. Cold finished	Stock
No. S17.....	Brass B	Free cutting, leaded brass. Annealed at 800 C.	Stock
No. S18.....	Alcoa 51S A	Alcoa 51S. Cold finished	Aluminum Company of America
No. S19.....	Alcoa 51S B	Alcoa 51S. Quenched in water from 970 F.	Aluminum Company of America
No. S20.....	Alcoa 51S C	Alcoa 51S. Quenched in water from 970 F. Held at 300 F. for 27 hr.	Aluminum Company of America
No. S21.....	Alcoa 51S D	Alcoa 51S. Annealed at 800 F.	Aluminum Company of America
No. S22.....	Alcoa 2S A	Alcoa 2S. Cold finished	Aluminum Company of America
No. S23.....	Alcoa 2S B	Alcoa 2S. Annealed at 600 F.	Aluminum Company of America
No. S24.....	Alcoa 2S C	Alcoa 2S. Annealed at 1100 F.	Aluminum Company of America
No. S25.....	Dowmetal A	Dowmetal A. Extruded	Dow Chemical Co.
No. S26.....	Dowmetal B	Dowmetal A. Quenched in water after 16 hr. at 750 F. Held at 350 F. for 22 hr.	Dow Chemical Co.
No. S27.....	Zinc A	Commercial zinc (99.95 per cent). Rolled	New Jersey Zinc Co.
No. S28.....	Zinc B	Commercial zinc (99.95 per cent). Annealed at 600 F.	New Jersey Zinc Co.
No. S29.....	Aluminum A	Alcoa 2S. Cold finished	Stock
No. S30.....	Aluminum B	Alcoa 2S. Annealed at 600 F.	Stock

Compression tests were made using a Riehle 50,000-lb. universal testing machine, and two Huggenberger type B strain gages set at 1-in. gage length and diametrically opposite on the specimen. The specimens were cylinders approximately 3½ in. long and ⅞ or 1 in. in diameter, with parallel milled ends. A spherical seat and hardened steel compression blocks were used. Tests were carried only to the limit of the strain gages, and yield strength values were calculated in the usual way from plotted stress-strain curves. The tests were run very slowly in the plastic range, allowing up to ten minutes for the specimen to approach equilibrium at each loading increment. No definite time interval was chosen, but each load was maintained until movement of the strain gages was imperceptible.

Torsion tests were made, using 6- to 8-in. gage lengths and $\frac{3}{4}$- to $\frac{7}{8}$-in. diameter bars. The testing machine used was a remodeled lathe to which a dead-weight lever arm loading device was attached to the tail-stock. A balanced loading arm was used extending 100 in. on either side of the center. The head stock was arranged to maintain the torque applied through the specimen and to allow controlled rotation in order to keep the loading arm balanced, as indicated by a spirit level. The initial loads were 30 lb. at the end of each arm, thus the nominal capacity of the machine was 6000 in-lb. With two exceptions all types of bars were strained well beyond their yield strengths in $\frac{3}{4}$- or $\frac{7}{8}$-in. diameter sections. The tests on cold-drawn monel metal and the hot-rolled 20 per cent chromium steel were completed in an Olsen 58,000 in-lb. torsion tester after the most essential data had been obtained in the dead-weight tester. The troptometer used in all torsion tests was a simple circular arc type having a curved scale placed 12 in. from the center of the specimen. A reading glass was used to read the scale to 0.01 in. The load could be maintained constant indefinitely in the dead-weight lever type torsion tester, and the tests were run slowly in the plastic range as in the compression tests.

In order to compare stress values in shear and compression, some means had to be selected for securing shear yield stresses comparable to compressive yield stresses. Because of the inherently different nature of shear and compressive strains, it is difficult to select any arbitrary torsional strain which will be directly comparable to a compressive strain such as 0.002 in. per inch. Neither is it reasonable to assume some new arbitrary shear strain, such as 0.002 in. per inch, of gage length, for the determination of a torsional yield strength; that is, provided the relationship between the actual strengths in shear and compression is to be established. (It should be remembered that such a shear strain represents 0.002 in. *tangential* movement with respect to a center 1 in. from the point of stress considered.)

In order to obtain a practical basis for making such comparisons, the known properties of mild steel were borrowed as a starting point. It is well known that a fairly constant relationship exists between the torsional and tensile (and therefore compressive) *yield points* of mild steel. Seely and Putnam[10] reported the ratio of the yield point stresses in shear and compression to be 0.65. (A somewhat lower ratio is obtained when tubular torsion specimens are used.) In order to obtain a relationship between allowable *strains* in shear and in compression, it is proposed that the corresponding elastic stress increments be related through a factor $\frac{S_s}{S_c} = 0.65$. The small angular deformation in torsion which corresponds to an elastic shear stress increment which is 0.65 times the allowable elastic compressive stress increment may then be used as an allowable *permanent* set in torsion, in exactly the same way as the compressive strain increment is used as the allowable *permanent* set in the established procedure for determining yield strength in compression.

A similar relationship might be set up with an $\frac{S_s}{S_c}$ ratio equal to unity, or equal to the theoretical value of 0.5

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[10] F. E. Seely and W. J. Putnam, "The Relation Between the Elastic Strengths of Steel in Tension, Compression and Shear," *Bulletin No. 115*, University of Illinois, Engineering Experiment Station (1919).

obtained from consideration of shear stresses developed in simple tensile or compressive members. However, the value 0.65 happens to be a better average for experimentally determined ratios of shear to compressive stress for the several ferrous and non-ferrous alloys used in this investigation; consequently, if a single formula is to be used for all metals, the ratio 0.65 has some merit.

The numerical expression for such an allowable angular set is readily found as follows.

Given:
E = modulus of elasticity in compression, in pounds per square inch,
E_s = modulus of elasticity in shear, in pounds per square inch,
r = radius of torsional test bar in inches,
J = polar moment of inertia $= \frac{\pi r^4}{2}$,
Δe_c = allowable permanent set at the compressive yield strength, in inches per inch of gage length, and
ΔS_c = elastic compressive stress corresponding to an elastic strain $= \Delta e_c$

Unknown:
$\Delta\Theta$ = unit angular deformation, corresponding to the allowable set in compression, in radians per inch of gage length of the torsional test bar,
ΔS_s = elastic shear stress corresponding to an elastic strain of $\Delta\Theta$
ΔM = increment of moment corresponding to $\Delta\Theta$, in inch-pounds, and
Δe_s = maximum tangential shear strain $= \Delta\Theta' \cdot r$, expressed in inches per inch of gage length of the torsional test bar. (This is the conventional unit shear strain.)

Calculation:

$$\Delta S_s = 0.65 \Delta S_c \text{ (assumed)}$$

$$\Delta M = \frac{\Delta S_s J}{r} = \frac{0.65\,\Delta\, S_c J}{r}$$

$$\Delta\Theta = \frac{\Delta M}{E_s J} = \frac{0.65\,\Delta\, S_c}{r E_s} = \frac{0.65\,\Delta\, e_c E}{r E_s} \ldots \quad (3)$$

In the case of a $\frac{7}{8}$-in. diameter steel torsion bar, the following values may be used as an example:

E = 30,000,000 lb. per sq. in.,
E_s = 11,500,000 lb. per sq. in.,
Δe_c = 0.002 in. per inch of gage length,
r = 0.4375 in., and

$$\Delta\Theta = \frac{0.65 \times 0.002 \times 30{,}000{,}000}{0.4375 \times 11{,}500{,}000},$$
$$= 0.00775 \text{ radians per inch of gage length,}$$
$$= 0.443 \text{ deg. per inch of gage length.}$$

If the torsional data are plotted as *M versus θ*, it will be easy to determine the moment and corresponding shear stress by a graphical construction similar to that now used for determining yield strength in tension or compression.

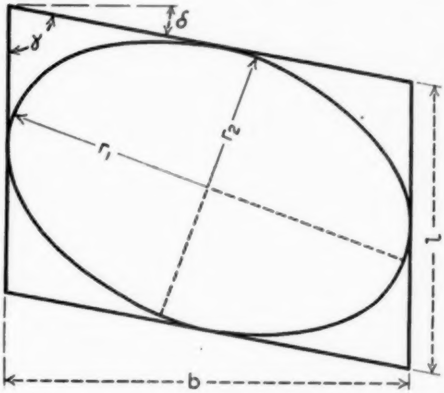


FIG. 5.—Cross-Section of a Sphere Inscribed in a Cube After the Cube Has Been Permanently Deformed.

It is of interest to note that if the allowable unit tangential shear strain, Δe_s, were taken as 0.002 in. per inch of gage length:

$$\Delta\Theta' = \frac{0.002}{0.4375} = 0.00457 \text{ radians per inch of gage length}$$
$$= 0.262 \text{ degrees per inch of gage length, or 59 per cent of the former value.}$$

Since the allowable set in torsion is less, the yield strength in shear will be less than that for the proposed method of calculation. The $\Delta S_s/\Delta S_c$ ratio corresponding to the above value would be only 0.383, which seems to be a relatively severe arbitrary limitation for shear stresses and strains.

Meyer hardness tests were made in the usual way, using the concentric loading method.[3] The time of application of each load was 3 min. for alloys of series Nos. 1 to 17, and 5 min. for all others. The Olsen hydraulic type Brinell tester used was calibrated by means of a proving ring.

The plastic deformation of the metal under the Brinell ball was studied by means of a split specimen held together in a clamp during application of the load on the Brinell ball. These specimens were carefully milled and lapped in such a way that flat, right angled, closely fitting halves were obtained. One of the contacting surfaces was marked with a sharp tool in a dividing engine, as illustrated in Figs. 7 and 8. The parts were then clamped together with sufficient force to prevent spreading when the Brinell load was applied, without, however, setting up undue elastic compressive stress in the specimen. It was found that the Brinell load required to produce a given size of impression was appreciably different than for a normal test. However, no appreciable difference was observed in the diameters of the impressions in the direction of, and normal to the compressive stress produced by the clamp; consequently, it was felt that the plastic strains were but little affected by the superimposed elastic compression.

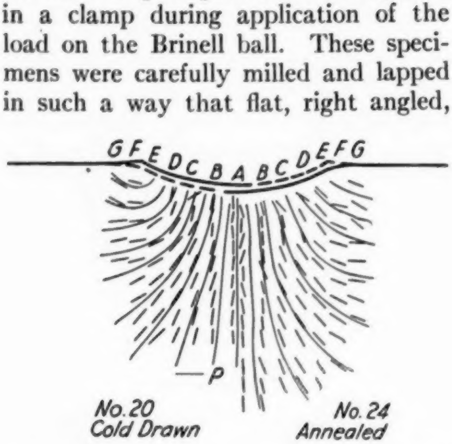


FIG. 6.—Showing the Method of Determining Stress Trajectories from the Directions of the Compressive Stresses at Several Points Along a Streamline.

The elevated surface contour corresponds to the data for the cold-drawn alloy at the left. The depressed surface corresponds to the data for the annealed state.

The cross-sections of the deformed specimens were photographed at three times magnification, and certain measurements of linear deformations were made from prints enlarged to ten times magnification (see Figs. 7 and 8). Other more detailed measurements of both lineal and angular deformations were made by direct observation of the ruled specimens at 100 magnifications.

It is believed that improved loading conditions would result if a true cylindrical test specimen were machined from the fitted halves. Such a specimen could be shrunk into an undersize steel ring so that essentially constant radial compressive stresses would result.

TEST DATA

The test data are summarized in Tables II, III, and IV and Figs. 4, 6, 7, and 8.

Much of the data obtained from the split hardness test specimens will be found in Table III and the curves on the page opposite. Since it was not practical to reproduce photographs of all of these specimens, the general type of impression was recorded in Table III, column D. Type A is represented by Fig. 7 and type C by Fig. 8. Type B is intermediate between A and C, in that the edge of the impression is neither sharp and raised well above the surface nor is it rounded and depressed. In type B the impression may show considerable general rise above the surface level, but its edge is not sharp at the points of contact with the ball as in type A.

Specimens Nos. 46 and 47 were impressed by a hardened steel cylinder

TABLE II.—PHYSICAL TEST DATA.

			A	B	C	D	E	F	G	H	I	J	K	L	M
Series	Alloy	Treatment	Meyer Hardness		L at $d = 5$ mm., kg.	Brinell Hardness	Proportional Limit, S_s, lb. per sq. in.	Yield Strengths			Ratios of S_s/S_c			E_s, lb. per sq. in.	E_c, lb. per sq. in.
			a	n				S_s at $\Delta e_c =$ 0.1 per cent, lb. per sq. in.	S_s at $\Delta e_c =$ 0.2 per cent, lb. per sq. in.	S_c at $\Delta e_c =$ 0.1 per cent, lb. per sq. in.	At Proportional Limit	At $\Delta e_c =$ 0.1 per cent	At $\Delta e_c =$ 0.2 per cent		
No. S1........	Armco A	Rolled	53.5	2.25	1995	96	20 850	23 400	23 550	35 150	0.65	0.66	0.67	11 650 000	28 470 000
No. S2........	Armco B	Annealed	41	2.39	1930	95	14 150	17 900	18 150	35 400	1.14	0.51	0.48	11 600 000	28 800 000
No. S3........	Mild steel A	Cold drawn	127	2.14	3960	190	33 200	54 800	.....	68 850	0.72	0.79	....	11 920 000	29 300 000
No. S4........	Mild steel B	Spheroidized	100	2.14	3120	149	18 650	33 600	35 200	40 050	1.27	0.84	0.85	11 900 000	29 550 000
No. S5........	Mild steel C	Quenched	130	2.17	4280	201	19 650	40 500	46 050	51 300	1.24	0.79	0.72	11 450 000	28 900 000
No. S6........	Mild steel D	Annealed	66	2.26	2505	120	24 900	25 800	25 850	34 700	0.92	0.74	0.74	11 650 000	28 870 000
No. S7........	S.A.E. No. 2330 A	Rolled	135	2.20	4640	216	22 200	43 830	46 550	65 700	0.54	0.67	0.68	11 200 000	29 420 000
No. S8........	20 per cent chromium A	Rolled	158	2.17	5210	242	27 150	78 000	.....	97 050	0.53	0.83	....	11 050 000	25 800 000
No. S9........	20 per cent chromium B	Annealed	114	2.26	4350	202	21 750	36 400	38 300	52 150	0.69	0.70	0.71	11 250 000	26 700 000
No. S10........	18 8 A	Rolled	111	2.34	4800	217	30 950	48 050	51 250	64 250	0.94	0.75	0.76	11 490 000	26 900 000
No. S11........	18 8 B	Annealed	68.5	2.37	3100	143	4 450	17 950	20 700	13 100	0.92	0.78	0.73	11 340 000	27 350 000
No. S12........	Monel A	Cold drawn	131	2.16	4280	201	45 900	72 700	75 900	81 950	1.03	0.89	....	9 630 000	25 400 000
No. S13........	Monel B	Annealed	71	2.26	2700	126	4 150	16 500	17 650	23 800	0.76	0.69	0.69	10 000 000	26 300 000
No. S14........	Herculoy A	Cold drawn	149	2.15	4760	226	34 830	67 250	77 450	64 000	1.18	1.05	1.05	6 170 000	14 020 000
No. S15........	Herculoy B	Annealed	34	2.44	2730	87	4 050	8 400	9 100	13 000	0.90	0.64	0.63	5 600 000	14 250 000
No. S16........	Brass A	Cold finished	88	2.12	2660	127	25 050	41 100	45 050	38 000	1.22	1.08	1.05	6 860 000	10 720 000
No. S17........	Brass B	Annealed	30	2.42	1470	74	4 950	8 800	9 100	12 250	0.61	0.72	0.73	5 050 000	14 350 000
No. S18........	Alcoa 51S A	Cold finished	35.5	2.10	1050	45	7 950	13 600	14 600	18 000	0.62	0.75	0.80	3 780 000	9 175 000
No. S19........	Alcoa 51S B	Quenched	46.5	2.16	1520	69	11 000	16 550	17 700	19 950	0.94	0.83	0.83	4 019 000	9 350 000
No. S20........	Alcoa 51S C	Precipitated	73	2.13	2260	103	21 200	30 500	32 300	44 550	0.68	0.68	....	3 775 000	9 675 000
No. S21........	Alcoa 51S D	Annealed	17.2	2.20	590	27	1 950	3 450	3 800	4 650	0.99	0.74	0.72	3 950 000	9 925 000
No. S22........	Alcoa 2S A	Cold finished	26.7	2.01	680	32	3 400	8 950	10 000	11 800	0.64	0.76	....	3 665 000	9 250 000
No. S23........	Alcoa 2S B	Annealed at 600 F.	13.2	2.23	475	22	1 350	1 900	2 150	2 400	1.21	0.79	....	3 665 000	10 175 000
No. S24........	Alcoa 2S C	Annealed at 1100 F.	13.5	2.20	470	22	1 150	1 800	2 100	2 370	0.99	0.75	....	3 560 000	10 200 000
No. S25........	Dowmetal A	Extruded	30	2.44	1500	61	4 350	7 600	9 000	22 830	0.30	0.34	0.39	2 300 000	6 060 000
No. S26........	Dowmetal B	Heat treated	28	2.49	1540	60	4 150	8 050	8 450	23 100	0.28	0.35	0.38	2 300 000	6 125 000

Columns A, B, C—See Eq. 1.
Column D—Series Nos. S1 to S17: Brinell $_{10/3000/180}$
Series Nos. S18 to S26: Brinell $_{10/500/300}$
Column E—Maximum shear stress at the proportional limit as determined by torsion tests of solid bars.
Column F—Maximum shear stress at a torsional yield strength based on an allowable set equivalent to $\Delta e_c = 0.001$ in. per inch, or 0.1 per cent, in compression.
Column G—Same as F based on $\Delta e_c = 0.002$ in. per inch, or 0.2 per cent.
Column H—Compressive yield strength based on an allowable set $\Delta e_c = 0.001$ in. per inch, or 0.1 per cent.
Column L—Modulus of elasticity in shear based on torsion tests of solid bars.
Column M—Modulus of elasticity in compression.
Column E—M—Data are based on the average of two torsion and two compression tests except in Series Nos. S4, S6, S15, S17, and S26 in which one torsion bar was used.

TABLE III.—DATA ON PLASTIC DEFORMATION IN THE BRINELL TEST.

Specimen	Series	Alloy	Treatment	A	B	C	D	E	F	G
				Diameter of Impression, mm.	Calculated or Normal Load, kg.	Increase in Load, per cent	Type of Impression	Rise Above Surface Level, mm.	Penetration, mm.	Meyer Hardness **n**
No. 2	No. S1	Armco	Rolled	5.20	2185	+9.4	A	0.10	6.0	2.25
No. 6	No. S2	Armco	Annealed	5.15	2050	+6.8	B	0.065	7.0	2.39
No. 8	No. S3	Mild steel	Cold drawn	5.10	4150	+0.5	A	0.105	6.5	2.14
No. 9	No. S4	Mild steel	Spheroidized	5.10	3270	−4.7	A	0.11	5.0	2.14
No. 10	No. S5	Mild steel	Quenched	5.10	4450	−7.4	A	0.125	6.0	2.17
No. 11	No. S6	Mild steel	Annealed	5.00	2510	+1.5	B	0.075	7.0	2.26
No. 12	No. S7	S.A.E. No. 2330	Rolled	5.15	4980	−8.8	A	0.085	7.0	2.20
No. 13	No. S8	20% chromium	Rolled	5.15	5560	+5.4	A	0.11	5.5	2.17
No. 14	No. S9	20% chromium	Annealed	5.05	4450	−5.6	B	0.07	6.5	2.26
No. 15	No. S10	18-8	Rolled	5.10	5000	+6.2	B	0.08	7.0	2.34
No. 16	No. S11	18-8	Annealed	5.25	3490	−9.0	C	0.025	9.5	2.37
No. 17	No. S12	Monel	Cold drawn	5.20	4650	+2.6	A	0.105	7.0	2.16
No. 18	No. S13	Monel	Annealed	5.10	2830	+3.5	C	0.01	9.0	2.26
No. 20	No. S14	Herculoy	Cold drawn	5.00	4760	−5.7	A	0.105	5.5	2.15
No. 24	No. S15	Herculoy	Annealed	5.10	1835	+6.5	C	0.02	10.5	2.44
No. 33	No. S16	Brass	Cold finished	5.00	2660	−4.8	A	0.095	5.5	2.12
No. 34	No. S17	Brass	Annealed	4.95	1440	+4.2	C	0.025	8.5	2.42
No. 35	No. S18	Alcoa 51S	Cold finished	5.40	1240	−4.0	A	0.19	5.5	2.10
No. 36	No. S19	Alcoa 51S	Quenched	5.15	1625	+10.8	B	0.07	6.5	2.16
No. 37	No. S20	Alcoa 51S	Precipitated	5.20	2445	+4.3	A	0.10	5.5	2.13
No. 38	No. S21	Alcoa 51S	Annealed	5.20	645	+8.5	B	0.06	8.0	2.20
No. 39	No. S22	Alcoa 2S	Cold finished	5.15	720	−2.8	A	0.17	4.25	2.01
No. 40	No. S23	Alcoa 2S	Annealed at 600 F.	5.25	535	+7.1	B	0.06	6.5	2.23
No. 41	No. S24	Alcoa 2S	Annealed at 1100 F.	5.25	520	+9.2	B	0.05	7.5	2.20
No. 42	No. S25	Dowmetal	Extruded	5.20	1650	+2.6	C	0.02	9.0	2.44
No. 43	No. S26	Dowmetal	Heat treated	5.15	1660	+2.0	C	0.04	8.0	2.49
VARIABLE IMPRESSION DIAMETERS										
No. 1	No. S1	Armco	Rolled	7.20	4550	−5.5	A	0.19	10.0	2.25
No. 2	No. S1	Armco	Rolled	5.20	2185	+9.4	A	0.10	6.0	2.25
No. 3	No. S1	Armco	Rolled	3.60	958	+4.9	A	0.045	5.0	2.25
No. 4	No. S1	Armco	Rolled	2.55	440	+9.8	A	0.025	2.5	2.25
No. 5	No. S2	Armco	Annealed	7.30	4720	−0.2	B	0.085	13.0	2.39
No. 6	No. S2	Armco	Annealed	5.15	2050	+6.8	B	0.065	7.0	2.39
No. 7	No. S2	Armco	Annealed	3.65	907	+2.9	B	0.060	3.0	2.39
No. 19	No. S14	Herculoy	Cold drawn	5.40	5590	−11.0	A	0.115	6.5	2.15
No. 20	No. S14	Herculoy	Cold drawn	5.00	4760	−5.7	A	0.105	5.5	2.15
No. 21	No. S14	Herculoy	Cold drawn	3.80	2660	+9.2	A	0.065	3.75	2.15
No. 22	No. S14	Herculoy	Cold drawn	2.70	1260	+15.0	A	0.03	2.75	2.15
No. 23	No. S15	Herculoy	Annealed at 800 C.	5.85	2560	+5.1	C	0.06	12.0	2.44
No. 24	No. S15	Herculoy	Annealed at 800 C.	5.10	1835	+6.5	C	0.02	10.5	2.44
No. 25	No. S15	Herculoy	Annealed at 800 C.	3.90	952	+10.3	C	0.02	7.0	2.44
No. 26	No. S15	Herculoy	Annealed at 800 C.	2.50	320	+8.1	C	0.02	3.25	2.44
VARIABLE ANNEALING TEMPERATURES										
No. 27	....	Herculoy	Annealed at 300 C.	5.05	4720	−4.7	A	0.105	5.0	2.13
No. 28	....	Herculoy	Annealed at 450 C.	5.00	3400	−1.2	A	0.09	6.5	2.17
No. 29	....	Herculoy	Annealed at 500 C.	5.15	3190	+3.3	A	0.085	7.0	2.22
No. 30	....	Herculoy	Annealed at 550 C.	5.10	2720	−0.1	B	0.07	7.5	2.26
No. 31	....	Herculoy	Annealed at 600 C.	5.00	2220	+2.0	B	0.06	8.0	2.30
No. 32	....	Herculoy	Annealed at 700 C.	5.10	1930	+1.3	C	0.025	9.5	2.38
No. 24	No. S15	Herculoy	Annealed at 800 C.	5.10	1835	+6.5	C	0.02	10.5	2.44
No. 44	No. S27	Zinc	Rolled	5.10	550	+2.2	C	0.045	8.5	2.27
No. 45	No. S28	Zinc	Annealed	5.10	530	+8.9	C	0.05	8.5	2.29
No. 46[a]	No. S29	Aluminum	Cold finished	5.20	......	......	C	0.04	10.5	[b]
No. 47[a]	No. S30	Aluminum	Annealed at 800 F.	5.30	......	......	D	0.00	10.5	[b]
No. 48	No. S29	Aluminum	Cold finished	5.10	......	......	A	0.17	4.5	[b]
No. 49	No. S30	Aluminum	Annealed at 800 F.	5.15	......	......	B	0.085	6.0	[b]

[a] 10-mm. diameter cylinder substituted for 10-mm. diameter ball.

[b] Meyer constants: Series No. S29, **a** = 29.7, **n** = 1.99; Series No. S30, **a** = 12.3, **n** = 2.28

Column A—Diameter of hardness impression as measured in the usual way with a low power microscope.

Column B—Calculated load required to produce given diameter of impression in normal, solid hardness specimen (Eq. 1).

Column C—Per cent increase in actual load applied over the calculated load.

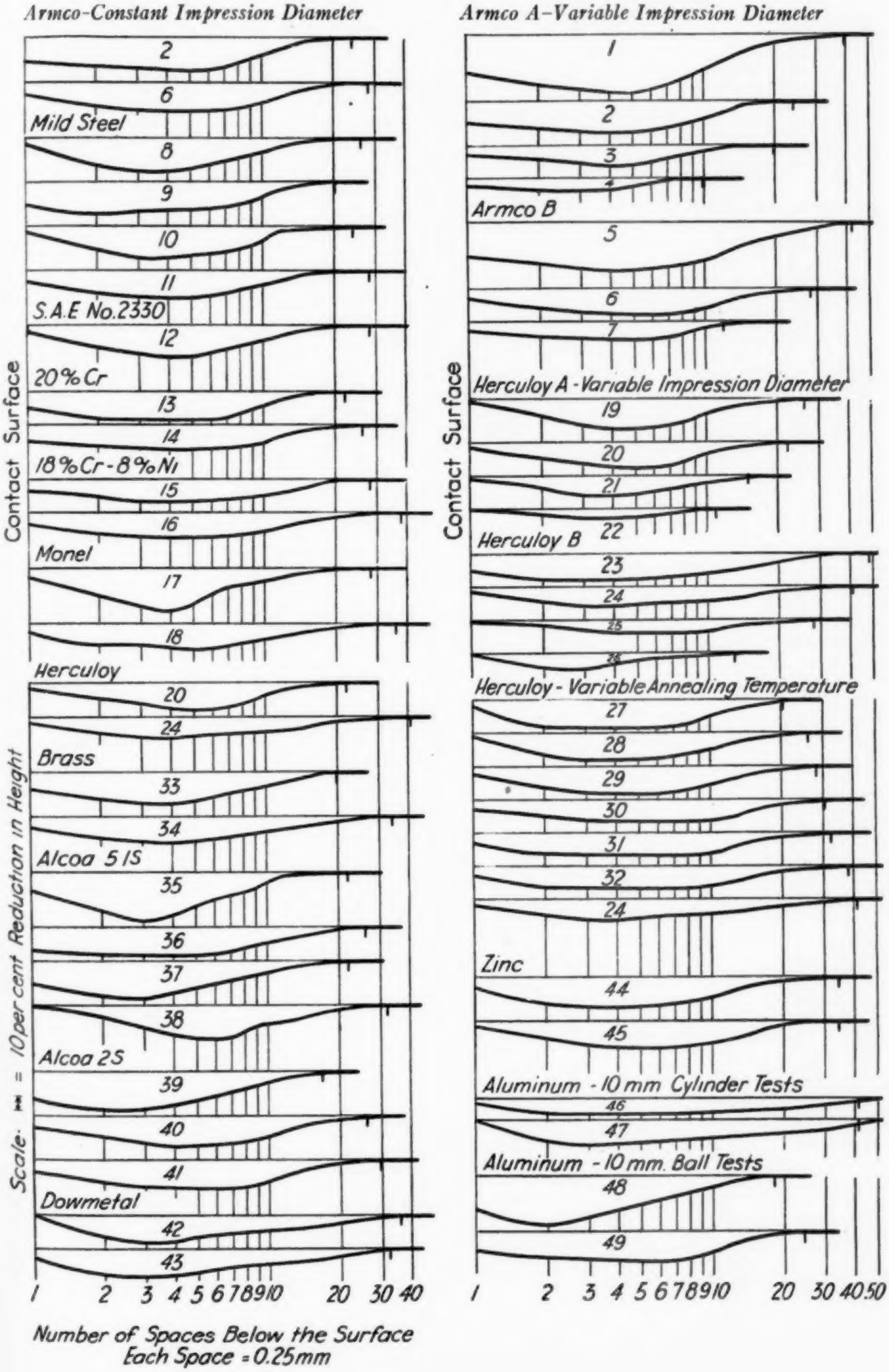
Column D—See discussion on pp. 126 and 132.

Column E—Maximum elevation of metal over the surface level at the edge of the indentation.

Column F—Distance below the surface to the first straight horizontal line, as estimated with a straight edge. Denoted by the short vertical lines on the curves opposite.

Column G—Meyer coefficient of strain-hardenability (see Eq. 1).

Per cent Compression at Various Depths Below the Surface



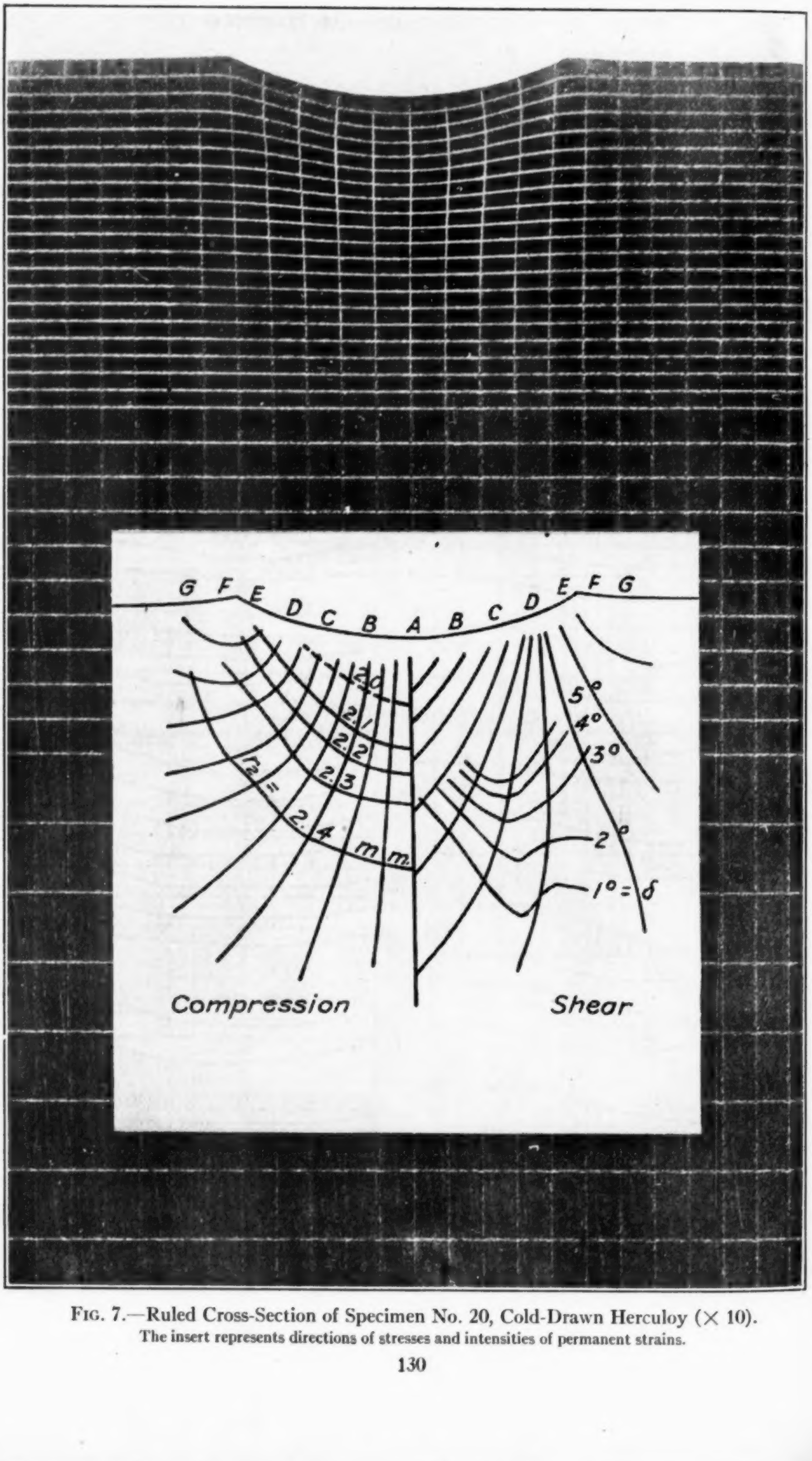


Fig. 7.—Ruled Cross-Section of Specimen No. 20, Cold-Drawn Herculoy (× 10).

The insert represents directions of stresses and intensities of permanent strains.

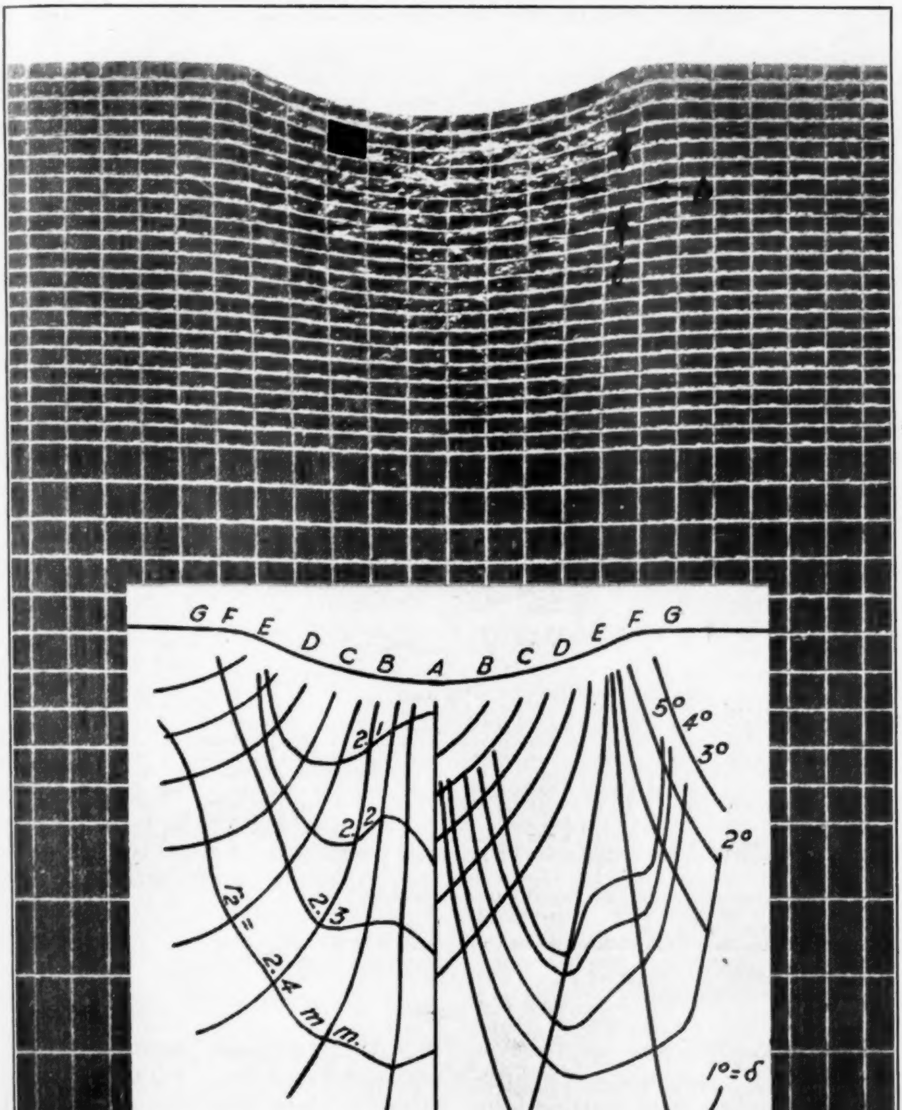


FIG. 8.—Ruled Cross-Section of Specimen No. 24, Herculoy Annealed at 800 C.
The insert represents directions of stresses and intensities of permanent strains.

10 mm. in diameter, the axis of the cylinder being normal to the ruled surface. The edge of the impression in specimen No. 47 was so highly curved and depressed that it was classified as type D.

Although only specimens Nos. 20, 22, 24, and 26 were analyzed for stress conditions under the ball, the curves opposite Table III, together with the data in columns D, E, and F, give some evidence of the characteristics of all of the impressions. The abscissas of these curves are the number of ruled divisions (spaced 0.25 mm. apart) below the surface of the specimen in the direction of the applied force, starting from the initial point of contact of the ball on the untested metal surface. The abscissas are plotted logarithmically in order to facilitate comparison of the more drastic deformations directly under the ball. The ordinates represent the reduction in height of each original rectangle, plotted to the scale indicated on the diagram.

TABLE IV.—CALCULATION OF DIRECTIONS AND INTENSITIES OF PLASTIC STRAINS ALONG STREAMLINE C OF SPECIMEN NO. 24.

A	B	C	D	E	F	G	H	I	J	K	L	M	N
	l mm.	b mm.	$\delta°$	$-\phi_l$	ϕ_r	$\tan\delta$	s, mm.	$\frac{d\phi_l}{ds}$	$\frac{-d\phi_r}{ds}$	$\frac{-d\tan\delta}{ds}$	$-\tan 2\alpha$	$-\alpha$	r_2, mm.
No. 1	0.432	0.545	10.6	0.146	0.086	0.187	0.43	−0.56	0.38	2.00	10.91	42°20′	0.207
No. 2	0.428	0.540	9.1	0.154	0.077	0.160	0.86	0.28	0.52	0.50	0.78	19°	0.204
No. 3	0.438	0.532	8.8	0.132	0.061	0.155	1.30	1.02	0.78	0.28	0.31	8°40′	0.212
No. 4	0.448	0.522	8.4	0.110	0.043	0.148	1.75	0.78	0.46	0.44	0.50	13°20′	0.214
No. 5	0.455	0.520	7.7	0.094	0.039	0.135	2.20	0.56	0.14	0.74	1.19	25°	0.220
No. 6	0.460	0.520	6.3	0.083	0.039	0.110	2.66	0.54	0.20	0.80	1.19	25°	0.224
No. 7	0.465	0.517	6.0	0.072	0.033	0.105	3.13	0.52	0.30	1.00	1.32	26°20′	0.226
No. 8	0.472	0.512	4.0	0.058	0.024	0.070	3.60	0.48	0.24	0.92	1.35	26°40′	0.233
No. 9	0.478	0.512	3.0	0.044	0.024	0.052	4.08	0.40	0.16	0.54	1.02	22°40′	0.237
No. 10	0.482	0.510	2.1	0.037	0.020	0.037	4.80	0.32	0.10	0.44	1.09	23°40′	0.240
No. 11	0.487	0.507	1.5	0.026	0.014	0.026	5.28	0.20	0.12	0.40	1.28	26°	0.242

Column A—Parallelogram numbers, from top to bottom of streamline C.
Columns B, C, D—See Figs. 5 and 8.
Columns E, F—See Eqs. 8 and 9.
Column H—Vertical distance from the top scratch to the bottom of the parallelogram.
Columns I, J, K—Slopes of the E *versus* H, F *versus* H, and G *versus* H curves.
Column L—Calculated from Eq. 10.
Column N—Calculated from Eq. 7.

This method of plotting reduced the sensitivity of the curves in the region far below the surface where the plastic deformations diminish to zero. However, in column F of Table III the depths of penetration are given as the distance below the surface to the first perfectly straight horizontal line. This determination was made with a steel straight edge placed on the photographs enlarged ten times, and although the change from a curved to a straight line is naturally very gradual, the same criterion of straightness was used in all cases, so the values given are reliable in a comparative way, and within absolute limits of ±0.5 mm. It should be noted that the impression diameters are approximately 5.1 mm. (5.0 to 5.25 mm.) except in those cases where the size of impression was purposely varied. No correction was applied to the various impression characteristics to compensate for this variation in diameter. In most cases the correction would be very small.

Specimens of cold-drawn and annealed Herculoy, specimens Nos. 20, 22, 24, and 26, were selected for more detailed analysis of plastic stresses and strains. Photographs, enlarged ten times, of the cross-sections of specimens Nos. 20 and

24 are shown in Figs. 7 and 8. The deformed squares were measured from the original specimen projected at 100 magnifications on the ground glass of a metallurgical microscope. These data were analyzed by the method reported in detail by Siebel,[11] a very brief account of which follows.

When an object is to be subjected to compressive and shear stresses, it may be split along a plane of symmetry without appreciably altering the stress conditions. This plane may be marked in such a way that subsequently produced plastic deformations will be measurable and subject to analytical treatment. Siebel's analyses are simplified by the assumption that the maximum shear theory governs elastic breakdown, and that the shear stress required for plastic flow is constant and equal to one-half the difference of the largest and smallest principal stresses, independent of the third principal stress. It has been shown that the maximum possible error due to this latter assumption is less than 15 per cent. The usual assumption is made that the volume change during plastic deformation is negligible.

Deformations in three directions at right angles are represented by the relations:

$$\phi_1 = \log_e \frac{r_1}{r_0} \quad \ldots\ldots\ldots (4)$$

$$\phi_2 = \log_e \frac{r_2}{r_0} \quad \ldots\ldots\ldots (5)$$

$$\phi_3 = \log_e \frac{r_3}{r_0} \quad \ldots\ldots\ldots (6)$$

where r_0 is the radius of a sphere which may be imagined to be inscribed in a small cube of the metal before deformation, and r_1, r_2, and r_3 are the principle radii of the inscribed ellipsoid resulting from permanent deformation of the sphere and cube.

If the axes of symmetry of the cube do not coincide with the principal stress directions, there will be shear deformations in addition to the tensile or compressive deformations of Eqs. 4, 5 and 6. In this case the axes of the ellipsoid will be inclined and unequal to the corresponding lengths of the resulting parallelopiped. A plane section of such an ellipsoid inscribed in the deformed cube is shown in Fig. 5. The length of the axis of greatest compressive deformation is given in Eq. 7.

$$r_2 = \sqrt{\frac{l^2 + b^2}{2} - \tfrac{1}{2}\sqrt{(l^2 + b^2)^2 - 4 \cdot l^2 b^2 \sin^2 \gamma}} \quad \ldots\ldots\ldots\ldots (7)$$

The cross-sections represented in Figs. 7 and 8 contained squares 0.50 mm. on a side before deformation. After deformation under the Brinell ball, certain vertical columns were designated as streamlines A to G, and detailed measurements were taken at 100 magnifications of all parallelograms in each streamline. The black parallelogram of Fig. 8 is C1, and those below it are C2, C3, etc. (The half space nearest the surface was not measured because it was not spaced at exactly 0.25 mm., and because the deformations near the surface were not homogeneous within a given parallelogram, especially in the region near the edge of the impression.) The directions of the principle compressive stresses were calculated for each parallelogram according to the method used by Siebel for analysis of wire drawing and extruding processes. Lengths l and b (see Figs. 5 and 8) and the average angles δ for streamline

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[11] E. Siebel, "The Plastic Forming of Metals," *Steel*, Vols. 93 and 94 (Eighteen installments from October 16, 1933 to May 7, 1934).

C of Fig. 8 are given in Table IV. Using the following equations for expressing deformations:

$$\phi_l = \log_e \frac{l}{l_o} \quad \ldots\ldots\ldots (8)$$

$$\phi_r = \log_e \frac{b}{b_o} \quad \ldots\ldots\ldots (9)$$

ϕ_l and ϕ_r were calculated and plotted against s. Values of tan δ were also plotted against s. Columns I, J, and K in Table IV are the slopes of these curves. The angle, α, was then calculated according to the following equation developed by Siebel and Huhne and given as Eq. 71 in Siebel's paper:[12]

$$\tan 2\alpha = \frac{\dfrac{d \tan \delta}{ds}}{\dfrac{d\phi_l}{ds} - \dfrac{d\phi_r}{ds}} - \tan \delta \,.\, (10)$$

where α is the angle which a principal direction of deformation makes with a streamline. These angles were plotted as short lines on a photograph of the cross-section, and the trajectories of deformation approximated from them as indicated in Fig. 6. Examination of Figs. 7 and 8 will indicate that much smaller squares would be necessary for an exact determination of stress directions in the region of high shear near the rim of the impression. Furthermore, it is believed that certain frictional effects at the surface may be responsible for a few unsatisfactory determinations of α in the first layer, and the trajectories were, in such cases, drawn perpendicular to the surface contour of the impression.

The directions of the stresses causing plastic deformation must coincide with the deformations; therefore, the compression lines of Fig. 6 and those at the left in Figs. 7 and 8 give an indication of the stress conditions under the ball during deformation. One set of principal shear lines is drawn in at the right of Figs. 7 and 8.

The cross lines at the left of Figs. 7 and 8 are the loci of points of equal strain, as determined by r_2 values. The cross lines at the right of Figs. 7 and 8 are loci of points of equal shear angles, δ.

DISCUSSION OF DATA

Surface Effects:

Several observations and analyses have been made of surface characteristics of Brinell impressions. It has been noted, for example, that soft annealed metals usually have "sinking-in" type impressions, and that cold-worked and hardened alloys tend to have sharp or ridging type impressions. Ichihara[13] and Krupkowski[9] made accurate measurements of the surfaces of typical types of impressions, and have derived equations for expressing the boundary curves. Norbury and Samuel[14] have shown that the extent of ridging or sinking-in is closely related to the work-hardening capacity as expressed by the Meyer **n** coefficient. The present experiments verify the fact that increasing elevation of the impression over the original surface accompanies decreasing values of Meyer **n**. In the Herculoy series, specimens Nos. 27 to 32, the change from a ridging to a sinking type of impression occurred upon annealing at 550 C. and higher temperatures. In a similar series of 30 per cent zinc brasses (not reported in detail here), the change occurred after annealing at 350 C. The **n** value is approximately 2.25 in both

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[12] E. Siebel, "The Plastic Forming of Metals," *Steel*, Vol. 94, p. 25 (1934).

[13] M. Ichihara, "A Contribution to Brinell Ball Hardness Tests," *Technical Reports*, Tohoku Imperial University, Vol. 10, p. 25 (1931).

[14] A. L. Norbury and T. Samuel, "The Recovery and Sinking-In or Piling-Up of Material in the Brinell Test, and the Effects of These Factors on the Correlation of the Brinell with Certain Other Hardness Tests," *Journal*, Iron and Steel Inst., Vol. 117, p. 673 (1928).

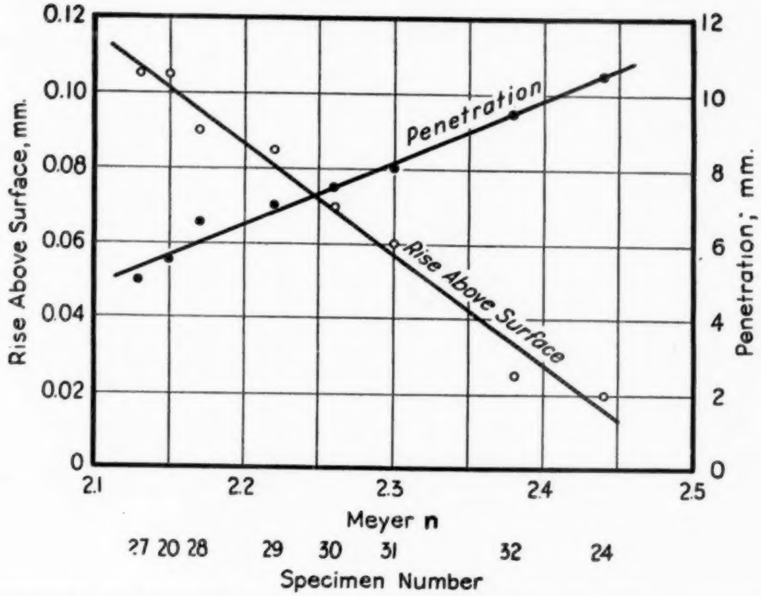

FIG. 9.—Showing the Relationships Between Penetration or Depth of Measurable Permanent Distortion, Rise Above the Surface, and Meyer **n** Coefficient for Cold-Drawn Herculoy Annealed at Various Temperatures.

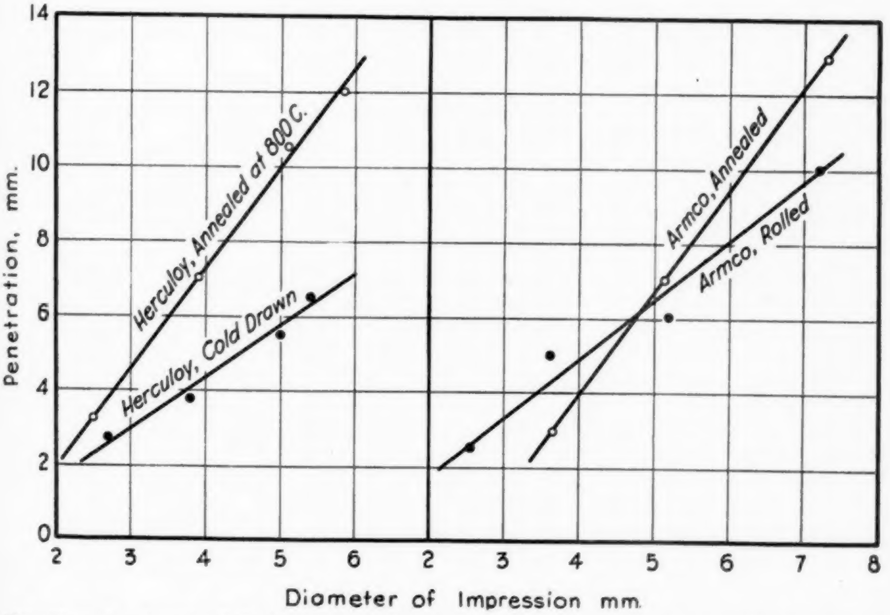

FIG. 10.—Showing the Relationship Between Penetration or Depth of Measurable Permanent Distortion, and the Diameter of the Impression.

cases. Norbury and Samuel's data indicate a change from ridging to sinking-in type impressions at **n** = 2.29. Thus, observation of the contour of the impression is of some value as an indication of the annealing temperature, or in other cases the amount of cold reduction, provided we know the characteristics of a given alloy after a given series of treatments. The data for the Herculoy annealing series is plotted in Fig. 9. The slope of the "rise" *versus* **n** line differs somewhat from the relationship found by Norbury and Samuel, probably because their criterion of "rise" differs from that used here.

If all the "rise" *versus* **n** relationships of Table III are plotted, it will be found that individual points may deviate considerably from the line of Fig. 9, but that the general trend is unmistakably in the same direction as the line. This will be found true of many of the possible relationships which could be plotted from the data of Tables II and III, and is probably to be expected considering the diversity of alloys and treatments included.

Penetration Effects and Thickness Requirements:

In the case of annealed Herculoy, the penetration or measurable depth of visible distortion increased lineally with the Meyer **n** coefficient. It is apparent from Fig. 9 that the depth of distortion in an annealed alloy may easily be twice as great as in a cold-worked alloy of the same composition. This is, of course, on the basis of equal impression diameters.

Data on the variation of penetration with size of impression is plotted in Fig. 10. The visible distortion varies lineally with the impression diameter for the metals tested. It is apparent that the rate of increase of penetration with diameter of impression is much greater for the annealed alloys than for the rolled or cold-drawn alloys of the same composition.

Considering the data summarized in Figs. 9 and 10, it is apparent that no simple limitation should be set up for minimum required thickness of hardness test specimens, since the depth of permanent deformation varies considerably with the work-hardenability. Although it is not claimed that the minimum thickness should equal the depth of penetration as described here, it seems reasonable to assume that the thicknesses should vary approximately as the penetration readings vary.

It is interesting to compare the results of direct determinations of minimum sample thickness with the above penetration values. Yamanouti[15] calculated stress distributions within Brinell test specimens and arrived at a ratio of six for thickness of specimen to depth of impression. Assuming a perfectly spherical impression, the depth corresponding to 5.1 mm. diameter is 0.7 mm., and the limiting thickness of specimen would be 4.2 mm. using Yamanouti's factor. This is less than any of the penetration values reported in Table III for comparable impression diameters.

Templin[16] made direct tests of required thickness for wrought aluminum alloys, and concluded that for the softer alloys the specimen thickness need be little more than twice the depth of the impression, and for the harder alloys no more than five times the depth. On a 5.1-mm. diameter basis, the specimen thicknesses would be 1.4 and 3.5 mm., even smaller than recommended by Yamanouti.

Hankins and Aldous[17] made tests on

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[15] H. Yamanouti, "Relation of the Size of the Test Specimens on the Brinell Hardness of Metals," *Journal*, Soc. Mechanical Engrs. (Japanese), Vol. 36, p. S86 (1933).

[16] R. L. Templin, "The Hardness Testing of Light Metals and Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part II, p. 283 (1935).

[17] G. A. Hankins and C. W. Aldous, "Minimum Dimensions of Test Samples for Brinell and Diamond Pyramid Hardness Tests," *Journal*, Inst. Metals (British), Vol. 54, p. 59 (1934).

copper, mild steel, and spring steels in various conditions and report required thickness ratios of 6 to 25. They indicate that the thickness ratio should vary with the material. However, the variations are not in the order which would be predicted from the present experiments.

Kenyon[18] made direct determinations of the required thickness of sheet metals for Rockwell hardness, "B" scale, tests. The smallest allowable thickness ratio reported was 6.

The A.S.T.M. standard method for Brinell hardness testing[19] requires a thickness ratio of 10, or a specimen 7.0 mm. thick in the case of a 5.1-mm. diameter impression. Although this ratio is undoubtedly adequate for most materials, it might be advisable to vary the minimum required thickness with the character of the impression.

Mechanism of Plastic Deformation:

Three independent investigations have established the fact that the Meyer **n** coefficient of strain-hardenability is closely related to the curvature of ordinary tensile or compressive stress-strain curves in the region of plastic deformation,[20, 21, 22] Kokado[20] and Schwarz[22] express the plastic stress-strain relationship as:

$$e = \alpha s^m \text{..........} (11)$$

or

$$s = \left(\frac{1}{\alpha}\right)^{\frac{1}{m}} e^{\frac{1}{m}} = (\text{constant}) \cdot e^{\frac{1}{m}}$$

which bears a close resemblance to Eq. 1. The exponent $\frac{1}{m}$ is the slope of the line obtained when stress s and strain e are plotted on logarithmic coordinates. This exponent was proved to bear a simple relationship to the corresponding hardness exponent **n** through the equation:

$$\mathbf{n} = 2\left(1 + \frac{1}{m}\right) \text{.......} (12)$$

These relationships were found to hold more exactly for deformations somewhat beyond the early stages of plasticity.

Montini[23] made a mathematical study of stress conditions at the surface of a Brinell impression. Kokado and Yamanouti used Eq. 11 and certain other assumptions to calculate stress distributions and intensities below the surface. Their results do not agree entirely with the stress conditions represented in Figs. 7 and 8. For example, according to their analysis the maximum strain is immediately below the ball, whereas the deformation curves opposite Table III indicate that the maximum plastic compression occurs well below the surface. It appears that the corresponding elastic condition established before permanent deformation begins is maintained to this extent during plastic deformation.

The superimposed impression contours of Fig. 6 illustrate the remarkable difference in the nature of ordinary Brinell impressions, a fact which may not be generally appreciated. The depth of visible distortion for specimen No. 20 is indicated at p, while the corresponding depth for specimen No. 24 is nearly twice as great. It is quite clear that the annealed metal has undergone very drastic deformation at much greater depths than has the cold-drawn metal. Furthermore, this deformation is largely due to

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[18] R. L. Kenyon, "Effect of Thickness on the Accuracy of Rockwell Hardness Tests on Thin Sheets," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 229 (1934).

[19] Standard Methods of Brinell Hardness Testing of Metallic Materials, (E 10 – 27), 1936 Book of A.S.T.M. Standards, Part I, p. 817.

[20] S. Kokado, "Hardness and Hardness Measurement," *Technology Reports*, Tohoku Imperial University, Vol. 6, p. 201 (1927).

[21] P. Ludwik, "Die Bedeutung des Gleit-und Reisswiderstandes für die Werkstoffprüfung," *Zeitschrift* des Vereines deutscher Ingenieure, Vol. 71, p. 1532 (1927).

[22] O. Schwarz, "Zugfestigkeit und Härte bei Metallen," *Zeitschrift* des Vereines deutscher Ingenieure, Vol. 73, p. 792 (1929).

[23] C. Montini, "Difetti della forma sferica e vantaggi di quella piramidale nella prova di durezza Brinell," *La Metallurgia Italiana*, Vol. 26, p. 172 (1934).

shearing action, as evidenced by the extreme depth at which $\delta = 1$ deg. in Fig. 8. The direction of maximum shear is apparently almost vertical along streamline *D*, as though a column somewhat smaller in diameter than the impression itself had been pressed into the block. It will be recalled that the r_2 deformations calculated by Eq. 7 are produced by combined shearing and compressive stresses. The peculiar shapes of the r_2 contours of Fig. 8 are due to high shearing deformations superimposed on the compressions.

In the case of the cold-worked specimen of Fig. 7, the underlying shearing deformations are much smaller, while the compressive deformations near the surface are higher. The dotted line of Fig. 7 represents values of $r_2 = 2.0$ mm. ($\phi_2 = -0.097$, see Eq. 5). These are higher deformations than found at any point in Fig. 8 and are produced almost entirely by direct compression. The high primary compression in this region is, of course, responsible for the lateral flow which produced the ridge at the contact surface.

The stress trajectories of specimens Nos. 22 and 26 were determined and gave essentially the same results as obtained for specimens Nos. 20 and 24. However, relatively few squares were deformed plastically because of the small impression diameters, and the final photographs are not reproduced here.

Effect of Shear and Compressive Strengths on Hardness Characteristics:

When this problem was in its early stages, S. C. Hollister, formerly of Purdue University, suggested that the fundamental cause of differences in the appearance of Brinell impressions might be found in the relative strengths of the metals under shear and compressive loads. Accordingly, torsion and compression tests were made of most of the alloys tested (see Table II). Comparative ratios of S_s/S_c were determined at the proportional limits and at values of $\Delta e_c = 0.05$, 0.1, 0.2, and 0.3 per cent. The proportional limit ratios were not significant in relationship to any of the hardness factors. The yield-strength ratios became quite constant for a given series at 0.1 per cent set and higher. The 0.1 and 0.2 per cent yield strength ratios are reported in Table II, columns J and K. Analysis of these data indicates a relatively high S_s/S_c ratio for the high ridging types of alloys, indicating that these alloys are relatively strong in shear, or that they tend to deform by compressive action in preference to shear. The alloys with sinking-in characteristics are correspondingly weak in shear and tend to deform by shearing action deep below the surface.

The average value of S_s/S_c at 0.1 per cent set is 0.85 for eight alloys which were definitely work-hardened by rolling or cold drawing. These alloys are characterized by type A or ridging impressions, by low **n** values, and by shallow penetrations. The corresponding average for these same alloys in the annealed state is 0.69. In this condition all of the alloys have type B or C impressions, higher **n** values, and deeper penetrations.

The above averages do not include 18 per cent chromium, 8 per cent nickel alloy, which has a remarkably high **n** value of 2.34 in the hot-rolled condition, and has yield strength ratios of 0.75 and 0.78 in the rolled and annealed conditions respectively. Although the strength values were greatly reduced by annealing, the **n** coefficient was increased only slightly. Furthermore, curves for specimens Nos. 15 and 16 opposite Table III show that the soft annealed 18-8 specimen underwent greater deformations than the rolled metal at all distances below the surface. Usually the rolled or cold-worked metal attained a higher deformation than the annealed metal at some distance below the surface, and then decreased more rapidly at greater distances below the surface.

The following summary, which includes all the tests for which yield strength data are available, gives further evidence of the general relationship of S_s/S_c to the type of impression:

Type A (12 specimens): $S_s/S_c = 0.81$ average and 0.66 to 1.08 range
Type B (8 specimens): $S_s/S_c = 0.74$ average and 0.51 to 0.83 range
Type C (6 specimens): $S_s/S_c = 0.59$ average and 0.34 to 0.78 range

The monel, Herculoy, and brass series show the greatest relative strengths in shear in the worked condition, the S_s/S_c ratios being greater than unity for the two copper alloys. Both Dowmetal series add confirmatory evidence to the relative strength theory. The heat treatment used produced some homogenization of the microstructure but did not change the physical properties appreciably. Both series show marked sinking-in characteristics accompanied by a remarkably low S_s/S_c ratio.

On the other hand, the cold-finished aluminum alloys have the most marked ridging characteristics without a correspondingly high S_s/S_c ratio. Thus it is possible that a relatively high shearing yield strength is a major contributing factor in producing ridging, but that other properties are also involved. As already indicated, the Meyer **n** coefficient of work-hardenability is definitely related to the surface conditions observed at Brinell impressions.

It is evident that more extensive tests covering many related conditions of heat and mechanical treatment should be carried out for each type of alloy to evaluate further the factors contributing to the complicated hardness test. It is suggested that, where possible, the torsional tests be made on hollow cylindrical specimens and that both the torsion and compression tests be carried to high deformations in order to evaluate the rates at which the alloys strain harden under compressive and shearing conditions. The ordinary conditions of elastic failure might then be extended well into the plastic region. Nadai and Davis[24] have very recently reported fundamental work along this line which should lead to many interesting applications.

It is the author's opinion that whenever plastic behavior is studied in forming operations, in testing of materials, or at points of stress concentration in structural or machine parts, the relative shear and tensile (or compressive) properties should be considered for their influence on the general character of the deformation.

The deformation curves for specimens Nos. 46 and 47 opposite Table III may be compared with the curves for specimens Nos. 48 and 49. It is apparent that the cylinder produces less surface compression but deeper underlying effects than a ball of the same diameter. It is undoubtedly true that the plane stress conditions used in photoelastic tests only approximate the true conditions under a sphere.

While the subject of correlation of hardness values with ultimate strength, percentage elongation, and similar properties was not considered in detail here, it was found that the relationship between Meyer **a** *versus* S_s, the yield strength in shear, was somewhat better than the **a** *versus* S_c relationship, but not sufficiently close to warrant the use of the hardness value as a substitute for the compressive or tensile value. A considerable amount of information of this type is available in work by Schwarz,[22]

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[24] A. Nadai and E. A. Davis, "Plastic Behavior of Metals in the Strain-Hardening Range," *Journal of Applied Physics*, Vol. 8, p. 205 (1937).

Townsend,[25] Rosenhain,[26] and others, and in O'Neill's book.[2]

SUMMARY AND CONCLUSIONS

1. The maximum compressive deformations occurred somewhat below the surface of the impressions rather than at the surface.

2. The maximum shearing deformations in silicon bronze specimens occurred along a cylindrical surface whose axis coincides with the direction of loading, and whose diameter is approximately 0.7 times the diameter of the impression.

3. The sinking-in types of hardness impressions are produced by relatively large underlying shear deformations. The ridging types of impressions are produced by relatively large surface compressions.

4. In most cases, the sinking-in types of impressions were associated with relative weakness in shear, as measured by the ratio of shear to compressive yield strength. The ridging types of impressions were associated with a high ratio of shear to compressive yield strength.

5. The alloy 18 per cent chromium, 8 per cent nickel was found to have an unusually high work-hardenability in the hot-rolled state, a strong tendency towards sinking-in type impressions, and other distinguishing characteristics.

6. The aluminum alloys, which tended to have ridging type impressions and low Meyer **n** coefficients, showed the poorest conformity to the general conclusion stated in item 4.

7. Dowmetal A was found to have an extremely low S_s/S_c ratio, a very high Meyer **n** coefficient, and a pronounced sinking-in type of impression, conforming to the general conclusion stated in item 4.

8. The copper-base alloys had very high S_s/S_c ratios in the cold-worked state, and conformed strictly to the proposed explanation of impression characteristics.

9. Direct measurements of plastic deformation in thick hardness test specimens indicated that permanent distortion occurred at a depth greater than some of the minimum thicknesses of hardness test specimens recommended in the past. It is possible that the practical effects of such underlying distortions on the measurements of the impression are negligible. However, if accurate requirements for minimum specimen thickness are to be set up, the type of impression (sinking or ridging) should be considered.

10. It was shown that the depth of visible distortion increased, and the rise of metal above the original surface decreased, as the Meyer **n** coefficient was increased by annealing cold-drawn Herculoy at progressively higher temperatures. These relationships are capable of rather general application.

11. The change from a ridging to a sinking type of impression occurs at about $\mathbf{n} = 2.25$ to 2.30.

12. The increase of depth of penetration with impression diameter was studied for Armco ingot iron and Herculoy. The rate of increase for the annealed state was nearly twice as great as for the unannealed state, which introduces another variable in determining minimum thicknesses of hardness test specimens.

13. The plastic deformations produced under a cylindrical indentor (load applied normal to the axis of the cylinder) indicated higher shearing stresses and deeper penetrations than occur under a ball.

14. A method was proposed for expressing a shear yield strength having an arbitrary but logical relationship to the tensile or compressive yield strength.

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[25] J. R. Townsend, "Relation of Hardness of Non-Ferrous Metals to Strength and Workability," *Metal Progress*, Vol. 26, p. 35 (1934).

[26] W. Rosenhain, "Brinell Hardness and Tensile Strength," *Metallurgist*, Vol. 7, p. 83 (1931).

Acknowledgment:

The author wishes to acknowledge the helpful guidance and encouragement offered by Dr. J. L. Bray, Head of Chemical Engineering, Purdue University.

The photoelastic analysis was made under the direction of Prof. S. C. Hollister, and his suggestions had much to do with establishing the course of this work. Mr. Reid L. Kenyon's suggestions on interpretation of results, and his assistance in preparing this material for presentation is deeply appreciated.

The author wishes to thank the Tinius Olsen Testing Machine Co. for loaning us a Brinell proving ring.

Mr. C. A. Reichelderfer, working under the National Youth Administration, and Mr. A. G. Cass, student, furnished valued assistance in measuring and calculating the stress trajectory samples.

The cooperation of the Aluminum Company of America, The American Rolling Mill Co., Crucible Steel Company of America, Dow Chemical Co., International Nickel Co., New Jersey Zinc Co., and Revere Copper and Brass Inc., in furnishing materials for these tests, is gratefully acknowledged.

DISCUSSION

Mr. R. L. Templin.[1]—The author has referred to some work previously reported before this Society.[2] It may be well to point out the probable reason why satisfactory Brinell hardness tests can be made on much thinner specimens than the author's rather involved analysis would indicate, in the case of some of the light metals and alloys. In the present paper, the specimens are of such thickness that the so-called "anvil" effect apparently does not come into play. All of the commercial Brinell testing machines use steel anvils for supporting the specimen. In the case of the light metals and alloys, using the thinner specimens, we undoubtedly get anvil effects, which as the author has suggested may be compensating, with the result that much thinner specimens than his analysis indicates, can be tested satisfactorily.

It is rather difficult to indicate just what all of the factors are which enter into the anvil effect. Even though they could be specifically enumerated, there would yet remain the question of individual quantitative effects. One of the factors would appear to be the coefficient of friction between the specimen and the anvil. Perhaps another is the moduli of elasticity of both the anvil and the specimen. Certainly we know that the coefficient of friction existing between the light metals and alloys and steel is relatively high, and thus in all probability, excessive distortion is prevented with the result that there is little, if any, measurable change in the Brinell hardness values when relatively thin specimens are tested.

Mr. V. E. Lysaght.[3]—I was particularly interested in the conclusion by the author that direct measurement of plastic deformation in thick specimens indicated that permanent distortion occurred at a depth greater than some of the minimum thicknesses of hardness test specimens recommended in the past. It is possible that the practical effect of such underlying distortions on the measurements of the impression are negligible.

It has been my experience that there is a tendency to test with a given load thinner material than should be tested, especially when equipment with lighter loads and equal sensitivity are available and are far more suitable and give more accurate results.

Mr. M. Gensamer.[4]—There is one other factor which nobody has mentioned which I think should be mentioned, and that is the rate of work hardening of the material, for the effects known as "sinking-in" and "piling-up" are best explained on this basis.

Mr. H. A. Schwartz[5] (*by letter*).—Since the material indented by the Brinell ball is severely cold worked it must be harder than in its original condition. The Brinell number thus

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[1] Chief Engineer of Tests, Aluminum Company of America, New Kensington, Pa.

[2] R. L. Templin, "The Hardness Testing of Light Metals and Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part II, p. 283 (1935).

[3] Wilson Mechanical Instrument Co., Inc., New York City.

[4] Assistant Professor of Metallurgy, Carnegie Institute of Technology, Pittsburgh, Pa.

[5] Manager of Research, National Malleable and Steel Castings Co., Cleveland, Ohio.

measures not the indentation hardness of the original material but that of the material hardened by the amount of cold work accompanying the particular penetration characteristic of the metal.

It was desired to gain some idea of the degree of hardening occurring under these conditions. Samples of ingot iron, 0.12 per cent carbon steel, 0.95 per cent carbon steel and malleable were accordingly tested by the following method:

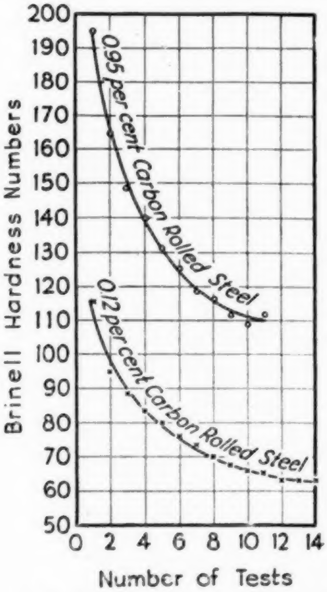

Fig. 1.—Effect of Strain Hardening During Brinell Tests 3000-kg. Pressure.

The material was first drawn 2 hr. at 650 C. and then tested for Brinell number. The heat treatment was repeated and the specimen returned to the machine in such a position that the ball would center in its old impression when the load was reapplied. The draw and test were repeated from ten to fourteen times, or until no further increase in diameter of impression could be observed.

Ingot and malleable iron are so soft that they require a reduced load (1000 kg.) to permit this test. The results are shown graphically in the accompanying Figs. 1 and 2.

It is interesting to note that with all four materials the unworked material is close to 0.6 as hard as by the usual form of test.

Mr. G. K. Dreher[6] (*by letter*).—While there are always numerous criticisms which might be made on any paper in which a variety of claims are set forth, we feel that the work is a good forerunner for a more detailed investigation of the various points covered either by Mr. Heyer or previous investigators.

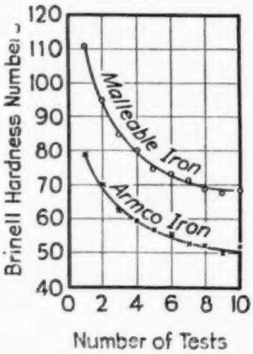

Fig. 2.—Effect of Strain Hardening During Brinell Tests, 1000-kg. Pressure.

It is our impression that considerably more work could be done, and that even some deductions might be drawn from the present investigation, in relation to the actual indentation of the Brinell test. Mr. Heyer makes a point of illustrating both the upraised impression boundary as well as the depressed boundary. The reading of these impressions, particularly those of the depressed type, frequently results in error. A standard or suitable compensation for reading this type of impression should be made.

Further, in working out relationships

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[6] Plant Manager, Ampco Metal, Inc., Milwaukee, Wis.

between shear strength and Brinell hardness, we feel that every effort should be made to specify accurately the material, its composition, and its structure in order to avoid the frequently misused conversion tables now in vogue whereby a tensile strength is given as approximating a Brinell result or *vice versa*. Much misunderstanding has developed along this line when engineers have attempted to use this conversion on materials other than those upon which the original tests were made.

MR. L. B. TUCKERMAN.[7]—I wish to commend the author on this most careful study of the type of deformation that takes place in the Brinell indentation test. I do not say hardness test. At the National Bureau of Standards those of us who have to use this test have agreed that it is probably better to avoid the use of the word "hardness" and call it an "indentation test," because we are not quite sure what is being measured. The author has shown us that the phenomena involved are much too complex to be represented by even the two coefficients of the Meyer formulation. Although he can say that on the average the ridging type of impression represents a higher shear strength, and on the average the depressed type of indentation represents a lower shearing strength, the ranges of the individual values overlap. In his type A, S_s/S_c gives a value as low as 0.66, and in his type C as high as 0.78; so that this difference in type of indentation involves something more than the S_s/S_c ratio. It would be welcome if somebody could find out what were the one or two or three or, perhaps, twenty other variables entering into the relation between the depth of the indentation and the load.

There is still too much of a tendency for people to think that there is such a thing as "real" or "true" hardness according to which all materials can be classified in a nice one-dimensional series, starting with "This material is the softest," "This material is harder than that material," and so on up to "This material is the hardest," with no inconsistencies, overlapping, or reversals of order. Those of us who have experimented with indentation tests, scratch tests, abrasion tests, and all the various tests by means of which some of the properties included under the term hardness are measured, know that that cannot be done unless the concept of hardness is narrowed beyond all recognition. It is welcome to find a person making careful study of one of these tests and learning some of the reasons why it cannot be done.

MR. R. H. HEYER[8].—Mr. Templin's experiments show that aluminum alloys may be tested in thinner sections than the conditions of plastic deformation in a thick specimen would indicate to be suitable. On the other hand, Mr. Lysaght has pointed out the tendency to use specimens that are too thin for a given hardness test. The complicated conditions of stress and the deep-seated deformations observed in the present tests are evidence that experimental determinations of limiting thicknesses should be made for each class of material and for each test condition if valid results are to be expected. In this way only may advantage be taken of the compensating factors which sometimes permit the use of lighter gage specimens than are ordinarily recommended. The etching method reported by Kenyon (see footnote reference 18 of paper) is recommended for this purpose.

Mr. Schwartz has attempted to measure the degree of work hardening

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[7] Assistant Chief, Division of Mechanics and Sound, National Bureau of Standards, Washington, D. C.

[8] Member, Research Dept., The American Rolling Mill Co., Middletown, Ohio.

occurring during the hardness test by a method involving successive applications of cold working and annealing. The annealing temperature used was high enough to promote recrystallization after cold working, therefore the final products were metallurgically different from the original specimens, which greatly reduces the significance of the test. By this method a cold-worked ingot iron would probably show a greater apparent work hardening in the hardness test than a corresponding annealed iron. The test does not separate the effect of previous cold work from that produced in the hardness test itself, and cannot be compared with the determination of the Meyer hardness constants as a means of evaluating work-hardening capacity.

Mr. Dreher expresses the need for a suitable compensation for readings of the depressed type. This implies that there is some correct type of hardness reading which is independent of the surface contour. It has been difficult to define any *correct* hardness in the past, and it is probably inadvisable to establish a single type of hardness determination as a criterion. It may be noted, however, that most hardness testers of the type which measure the depth of penetration use the original surface as a reference, disregarding both "piling-up" and "sinking-in."

The author agrees with Mr. Tuckerman that there may be twenty other variables involved. It now appears that the hardness indentation represents just as complicated a situation as the fracture of the standard round tension test specimen.

Mr. Gensamer stated that "sinking-in" and "piling-up" are best explained on the basis of the rate of work hardening. Reference was made to experimental data on this subject by Norbury and Samuel (see reference to footnote 14), and in Fig. 9 additional evidence was presented showing the relationship between the Meyer work hardening coefficient and "piling-up" in the hardness test. This relationship is, however, difficult to explain on the basis of work hardening effects alone. Why do metals which are not readily work hardened during testing (because of prior cold working) tend to "pile-up" around the ball? On the basis of the present determinations of shear and compressive properties it is indicated that in such cases the resistance to plastic deformation in shear has been built up to a larger extent (by the prior cold working) than has the resistance to compression; therefore, the compressive deformations are most prominent in the hardness test and cause extrusion of metal around the ball at the surface of the impression.

THE STIFFNESS OR FLEXURE TEST

By H. L. MacBride[1]

Synopsis

The paper discusses the test known as the cantilever bend, stiffness, or flexure test for light wire, sheet, and strip of various materials. A description is given of a stiffness testing machine (the Tour-Marshall tester) and the method of testing explained. Charts are shown of typical tests on different materials and a brief interpretation given, with a comparison of stiffness test results with those given by other methods.

Introduction

The stiffness test has a broad field of usefulness in comparing the elastic and plastic qualities of materials where the available specimens are very thin, soft or brittle. The difficulties encountered in making reliable tension, ductility and hardness tests on such specimens are well known, as for example on fine wire, thin or soft sheet and strip, thin plastic, ceramic, and fibrous compositions.

Some of the advantages of the method of test described in this paper are as follows:

1. Specimens are easily prepared. Wires, ribbons, and narrow strips, ordinarily received in coils, are tested without straightening and thus introducing variable amounts of cold work. Sheets are merely sheared roughly to shape and then notched in the manner discussed later in this paper.
2. The test is consistent in that tests on like samples can be depended upon to repeat within 1 per cent of the load reading.
3. The test is sensitive in detecting small variations in elastic and plastic qualities.
4. In many cases the straining action of the test closely approximates the demands upon the material in service.
5. The test is quickly and easily made and requires little skill on the part of the operator.

Description of the Stiffness Testing Machine

The instrument used in making the tests discussed in this paper was the Tour-Marshall motor-driven machine of 40 in-lb. bending moment capacity, as shown in Fig. 1. This machine operates on the cantilever-bend principle. A specimen is shown clamped in the vise, which is turned about a central spindle, either by hand, using the crank shown at the left, or by motor through gearing. The lever for engaging and disengaging the motor is just below the hand crank. Cantilever bending of the specimen is obtained by rotating the vise in a counter-clockwise direction, pressing the free end of the specimen against the under side of the roller pin, shown projecting from the dial face. This dial is part of the pendulum, which is free to swing on ball bearings concentric with the rotating vise. Different capacity ranges are obtained by placing different

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[1] Research Engineer, Tinius Olsen Testing Machine Co., Philadelphia, Pa.

weights on the pendulum pin. Engraved on the dial face is the angular deflection scale, reading from 0 to 90 deg. The deflection pointer moves with the vise, to which it is secured by a friction or slip collar, so that adjustment to zero can be made at the start of the test in the event that the specimen is not exactly straight. The deflection reading, in degrees, is the angle included between the original position of the roller bending pin, the edge of the vise, which is on the center of rotation, and the position of the bending pin after load is applied and the specimen bent. Load is indicated on the upper scale, fixed to the housing, the graduations on this scale being from zero, when the pendulum is at rest, to 100 per cent, when the pendulum has been lifted by the reaction of the specimen to a horizontal position. The pointer for the load scale is a mark engraved on the pendulum dial plate, since load depends only upon the angle through which the pendulum has been deflected from the vertical. Load is, in fact, proportional to the sine of this angle and the load scale is so graduated that the 10 per cent mark is at an angle whose sine is 0.10, 40 per cent is at an angle whose sine is 0.40, etc. The load scale can be shifted slightly before starting the test to bring its zero in line with the pendulum pointer in the event that the machine is not quite level.

A contact light is provided which, with metallic specimens, indicates when the specimen first touches the bending pin. It is useful in obtaining an accurate zero setting of load and deflection at the start of the test and also for indicating the point where load is removed from the specimen when the machine is reversed to find the "set angle."

The motor drive rotates the vise and applies load at a slow constant rate, an aid in obtaining uniform results on certain kinds of material. Vibration of the motor, installed inside the housing on a loosely hinged mounting, is utilized to minimize friction in the pendulum bearings.

METHOD OF TESTING

After starting the motor the specimen is clamped firmly in the vise with the center line approximately parallel to the face of the dial plate. Round samples should be lined up so that they will lie in the hollow face of the bending pin roller while being tested. The roller should contact flat specimens in the center to avoid twisting and to equalize any twist existing in the specimen before bending. If specimens are already slightly curved it has been found best to place them in the vise convex side up, so that the test bend is a continuation of the initial bend. Needless to say, samples should not be straightened before testing, unless it is desired to determine the effect of such straightening on the material.

Using the hand crank the vise is rotated, bringing the specimen up against the under side of the roller bending pin. When contact is just made, the light, set in the housing to the right of the dial plate, will start flickering on and off. The deflection pointer is then adjusted to indicate zero angle.

The motor-engaging lever is held down until the test is completed, at 90 deg. bend, until fracture of the specimen, or until the bend angle or load decided upon for the test is reached. In the various tests charted in this paper, full 90-deg. bends were made in order to get a more complete picture of the elastic and plastic qualities of the samples, but smaller angles of bend may give all the information that is necessary in many cases. After making the specified bend, the motor lever is released and the vise backed up quickly, using the hand crank. When the load indication has again become zero and the light goes out the "set angle" resulting from the bend is indicated by the deflection pointer.

The number of readings taken and whether or not a stress-strain diagram is constructed depends, of course, upon how much information is required for comparison and classification of the material in question. Brittle compositions may have their production and acceptance control based simply upon ability to support a specified minimum load at some given angle, or to bend through some specified minimum angle without fracture. More ductile materials may be classified as to strength and hardness by specifying the loads corresponding to certain bend angles and by limiting the permissible permanent set after some given bend angle.

The amount of weight to use on the pendulum is determined by trial, but the range of the machine for any one weighting is sufficient to cover any ordinary variations found in specimens of like material and gage. The weight used, times the pendulum length (4 in. on the machine shown in Fig. 1) gives the maximum bending moment applied to the specimen when the pendulum has been lifted to a horizontal position. The load scale reading is then 100 per cent.

Two positions of the bending pin are provided on the pendulum dial plate, giving a bending span of either 2 or 4 in. An attachment, not shown, gives a 1-in. span. The shortest span is used that will give sufficient bend with the weights provided.

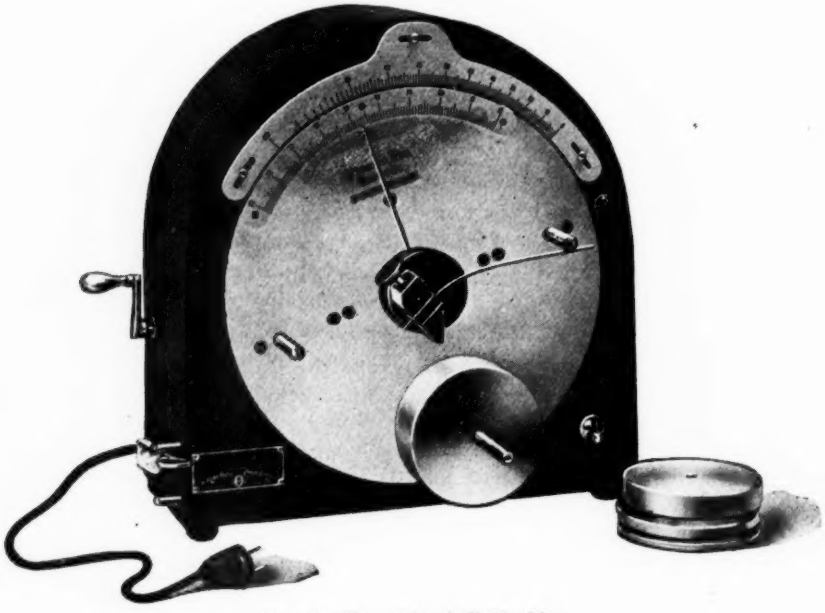


Fig. 1.—Tour-Marshall Machine.

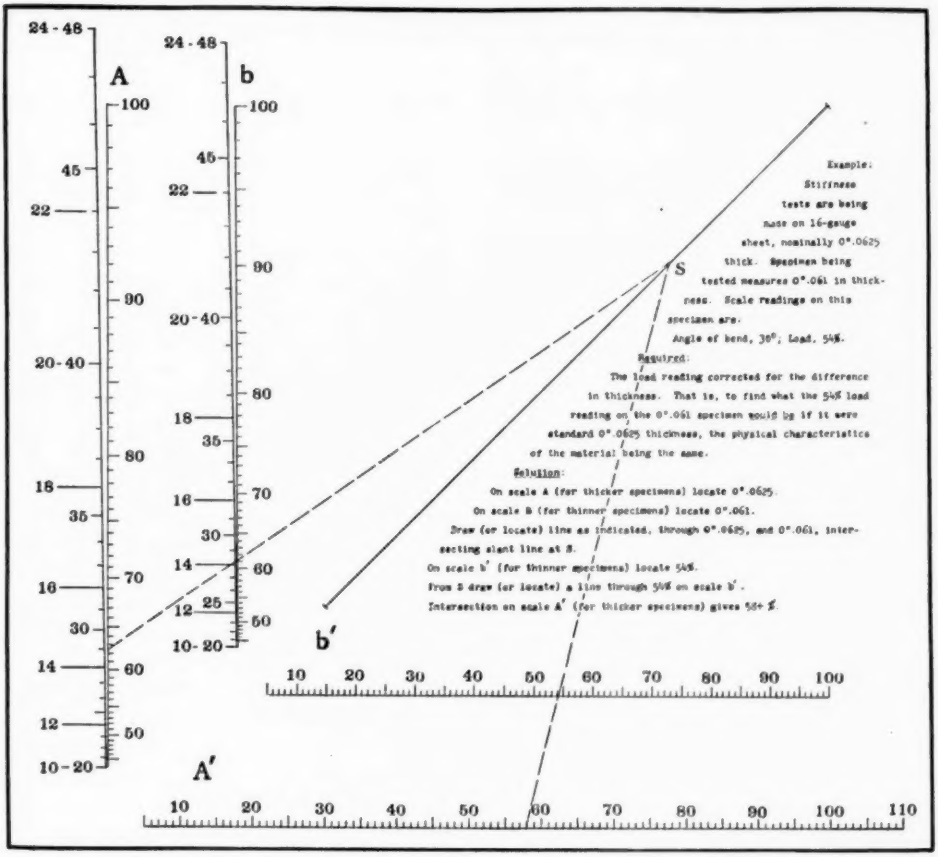
On the Tour-Marshall tester the load in pounds is

$$W = \frac{0.04BP}{L}$$

where B = the observed load scale reading,
P = the pendulum weight, and
L = the span in inches.

The expression "2-90 stiffness" refers to the load necessary to bend the specimen 90 deg. when applied 2 in. from the vise.

In flat rectangular sections I varies as the width and cube of the thickness. Load W, therefore, for a given deflection will vary with the cube of the thickness, other factors being constant. In round specimens the load for given deflection will vary with the fourth power of the diameter.



Example:

Stiffness tests are being made on 16-gauge sheet, nominally 0".0625 thick. Specimen being tested measures 0".061 in thickness. Scale readings on this specimen are.

Angle of bend, 30°; Load, 54%.

Required:

The load reading corrected for the difference in thickness. That is, to find what the 54% load reading on the 0".061 specimen would be if it were standard 0".0625 thickness, the physical characteristics of the material being the same.

Solution:

On scale A (for thicker specimens) locate 0".0625.

On scale B (for thinner specimens) locate 0".061.

Draw (or locate) line as indicated, through 0".0625, and 0".061, intersecting slant line at S.

On scale b′ (for thinner specimens) locate 54%.

From S draw (or locate) a line through 54% on scale b′.

Intersection on scale A′ (for thicker specimens) gives 58+ %.

Fig. 2.—Correction Chart for Thickness Variations, Flat Specimens.

Load for a given small elastic deflection in a cantilever is computed by the standard formula

$$W = \frac{3EID}{L^3}$$

where E = the modulus of elasticity,
I = the section moment of inertia about the neutral plane, and
D = the deflection at the loaded end.

Tests made on plastic and metal specimens cut from the same pieces, but differing slightly in thickness showed that the load for the same deflection did vary practically as the cube of the thickness, not only in the elastic but throughout the plastic portion of the stress-strain curve. Annealed round steel wire showed the expected fourth-power relation.

An explanation of the fact that the elastic bend theory holds true—under the conditions of the stiffness test—for plastic bending is that the straining can be considered a succession of infinitely small elastic bends, the modulus of elasticity being different, after the proportional limit is passed, for each bend. The observed load at any deflection is an integral of the previous load increments.

In testing sheet or strip for temper and comparative strength a correction must be made for thickness variations. An alignment chart, as shown in Fig. 2, based on the cube-of-thickness relation, has been prepared for convenience in making the correction without computation. It is intended for use in correcting for thickness variations of 10 per cent or less. Figure 7 shows typical test results before and after correction.

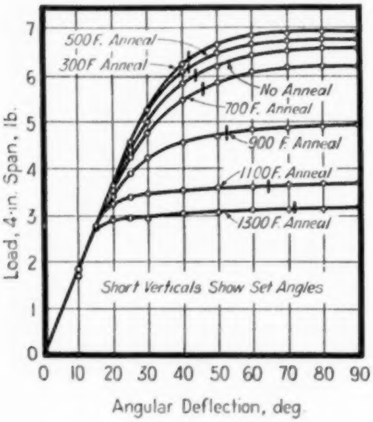


Fig. 3.—0.082-in. Music Wire.

In testing wire and narrow strip, where the specimen represents the actual shape of the material in use no correction need be made for thickness variations from gage. Small variations will not perceptibly affect the shape of the curve or the set angle.

In the test of specimens cut from sheet metal where conclusive information is required regarding its suitability for drawing and forming operations, in other words where it is desired to investigate the plastic rather than the elastic qualities, it appears advisable to confine the bend to one particular section of the specimen. The plane of maximum stress in a cantilever bend test is that cross-section nearest the vise. A specimen first bends elastically along its entire length, the bend being greatest nearest the fixed end. When the elastic limit of bending is reached yield takes place in the outer fibers of that section. Cold-work strengthening follows, so that, in some metals, the yielded section may quickly become stiffer than it was before the yield. The effect is to some extent the same as if the jaws of the vise were gradually extended to shorten the bending span. The plastic bending element moves away from the vise so that the test is actually a succession of partial tests. It is obviously desirable that a true picture be given of the entire elastic and plastic behavior of the same particles, rather than such a vague composite.

To confine the bend, various close-clamping methods were attempted but discarded because unavoidable small variations in such short spans caused wide fluctuations in results. It also seemed necessary to clamp the specimens very tightly in order to prevent bends from creeping back under the jaws, the heavy pressure causing other errors due to flattening or biting into the softer materials.

A notched specimen, of the shape outlined in Fig. 11, was finally adopted and seems to solve the problem. Specimens are easily prepared, a number being clamped together and notched with a hack saw, the notches then being smoothed with a thin file. The specimen is clamped with the center of the notch at the edge of the vise.

STIFFNESS TESTS ON WIRE

Figure 3 shows stress-strain diagrams of tests made on seven samples of high-carbon music wire of 0.082 in. actual diameter. The diagrams give a clear picture of "heat aging" and low-temperature hardening on cold-worked steel. A length of wire was cut from the coil and run through a straightening machine. This device bends the wire well beyond its elastic limit, the bend angle being rotated at high speed about the axis of the wire as it is pulled through the machine. The result is that the wire is strained to a nearly plastic state and straightened by the tension necessary to pull it through. Aging gradually restores the elasticity, which process may be accelerated and strains removed by a low-temperature anneal.

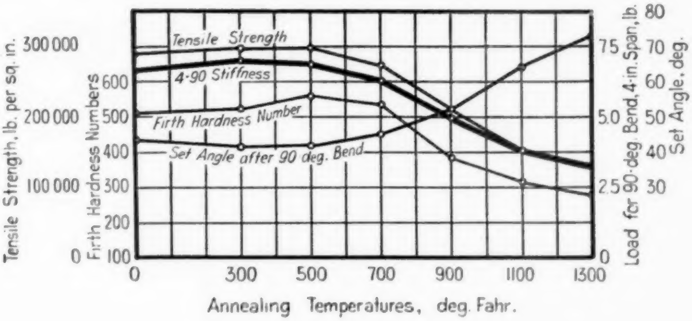


FIG. 4.—0.082-in. Music Wire.

After straightening, the wire was cut into 8-in. lengths and separated into seven lots. These were given different annealing treatments as follows: no anneal; 300 F. for ½ hr., cooled in 1 hr.; 500 F. same; 700 F. same; 900 F. same; 1100 F. same; and 1300 F. same. All samples were aged about one month before any tests were made.

The curves in Fig. 3 show the effect of this mild heat treatment. The no-anneal wire had a 4–90 stiffness of 6.6 lb. Its elastic limit in bending was 24 deg. The wire annealed at 300 F. had a 4–90 stiffness of 7.0 lb. and the elastic limit was increased to 26 deg. At 500 F. the wire showed a slight decrease in 4–90 stiffness and also a lower elastic limit. At 700 F. the stiffness curves showed the anneal quite clearly. The tests on the higher-temperature anneals showed the gradual flattening out of the stress-strain curve and a decreasing radius of the curve as it passed the elastic limit. The shape of the curve at this point was apparently characteristic of the internal structure of the material, since it has been observed that wires of

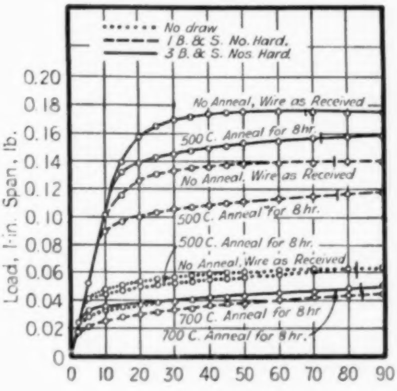


FIG. 5.—0.020-in. Grade A Nickel Wire.

the same composition and heat treatment give similar yield curvature, regardless of differences in diameter. The short vertical marks on the curves denote the angle of permanent set after the load was removed. This angle is read from the deflection dial and, with the 90-deg. stiffness reading, seems sufficient to classify and compare wire, at least for the purpose of insuring uniformity in elasticity and strength.

In Fig. 4 the 4–90 stiffness of the seven samples is compared with tensile strength and hardness tests made on specimens from the same piece with the same annealing cycles. The close and consistent agreement of all three tests is apparent. Each curve is the average of three tests.

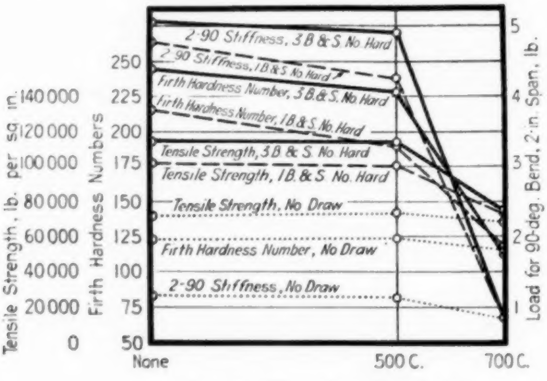


Fig. 6.—0.079-in. Grade A Nickel Wire.

Figure 5 records tests on specimens of grade A nickel wire 0.020 in. in diameter, made in connection with the work of Subcommittee IV on Mechanical Tests of the Society's Committee B-4 on Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys. Samples were supplied and annealed by the committee, in nine states of temper and anneal, as shown on the chart. The specimens were annealed at 500 and 700 C. for 8 hr., followed by cooling at the rate of 50 deg. Cent. per hour. All samples were straightened before annealing by stretching 1 per cent.

The height of the load ordinates, the slope of the plastic portion of these curves, and the radius of curvature as the curve leaves the elastic line show quite clearly the effect of the annealing treatments. On the soft "no draw" wire, the 500 and 700 C. anneals made little change in the already large-grained structure, except to relieve the straightening strains, and little change in stiffness characteristics. The 1 B. & S. No. hard wire was stiffer. The 700 C. anneal practically removed all traces of cold work from all three tempers. Note the almost precisely similar shape in the curves showing the same anneal on different draw hardnesses.

Figure 6 compares the 2–90 stiffness of grade A nickel wire with hardness and tensile strength. The samples were too short for reliable elongation measurements. The wire, of 0.079-in. diameter, was of the same tempers and annealing treatments as the 0.020-in. wire covered by Fig. 5. The 0.020-in. wire was too small for hardness tests, but it is logical that the close agreement in stiffness.

hardness, and tensile strength found for the 0.079-in. wire would apply in the case of the lighter wire.

Stiffness Tests on Sheet Metal

In determining the drawing and forming qualities of sheet metal the Tour-Marshall tester affords information regarding the elasticity, toughness, spring back, plastic flow, work-strengthening characteristics and hardness. It is true that much of the data is comparative and that absolute values, gaging one kind of material against another, are not easily determined. However, each type of drawing and forming operation seems to require its own more-or-less arbitrary set of standards, based upon practical experience.

It would appear, then, that the consideration of what units to use in expressing these arbitrary standards is not of great importance. It is, of course, essential that the test employed give a true indication of the qualities actually required in the material for satisfactory use, and detect small variations in such qualities.

Figure 7 shows stiffness tests on a commercial brass strip. Seven samples were tested, representing cold-rolling reductions of from 10 to 65 per cent, the pieces having been first fully annealed. The upper set of curves are stress-strain diagrams from the observed angle and load readings. Specimens varied from 0.0135 to 0.0146 in. in thickness. Corrections for these thickness variations were made, based on a standard of 0.0135 in. The lower set of curves shows the result of correction. The shape of the curves, the slope of the plastic portion and the radius of curvature after yield, show little change. The load ordinates, however, now give a true picture of the variations in temper. The sketch shows the actual shape of the specimens used.

Figure 8 compares the 1–90 stiffness and set angle values from the tests charted in Fig. 7 with hardness and tensile strength results on the same specimens. There was no elongation reading obtainable from tension tests of the higher-tempered specimens, due to rebound. Indentation hardness tests required piling of specimens to avoid "anvil effect" and their reliability is accordingly open to question.

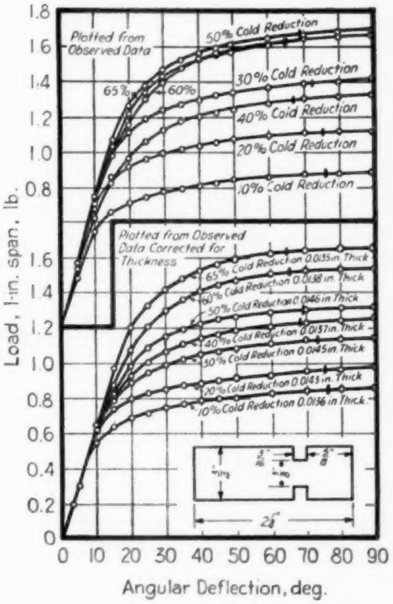


Fig. 7.—67 per cent Copper, 33 per cent Zinc Commercial Brass Strip.

Figure 9 records stiffness tests of specimens from the same reel of 0.027 by $\frac{3}{8}$-in. cartridge brass. Various anneals were given, resulting in the grain sizes shown on the chart. The curves show quite clearly the effect on elasticity and strength of this heat treatment. The set angle readings decrease regularly with grain size and there is the characteristic change in yield curvature. Tension test results on this strip as compared to the 2–90 stiffness are shown in Fig. 10. Note the agreement of "set angle"

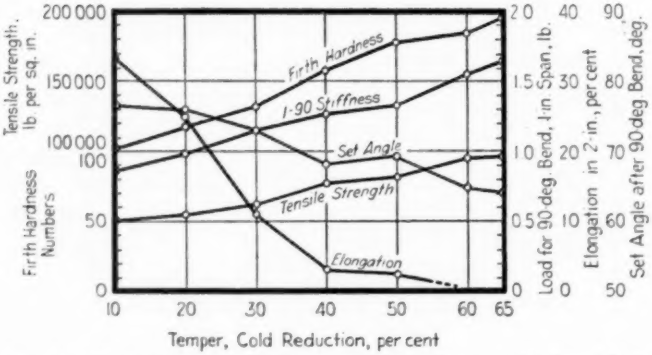


FIG. 8.—67 per cent Copper, 33 per cent Zinc Commercial Brass Strip.

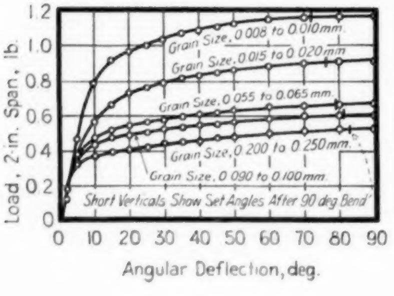


FIG. 9.—0.027 by $\frac{3}{8}$-in. 70 per cent Copper, 30 per cent Zinc Brass Strip—Various Anneals.

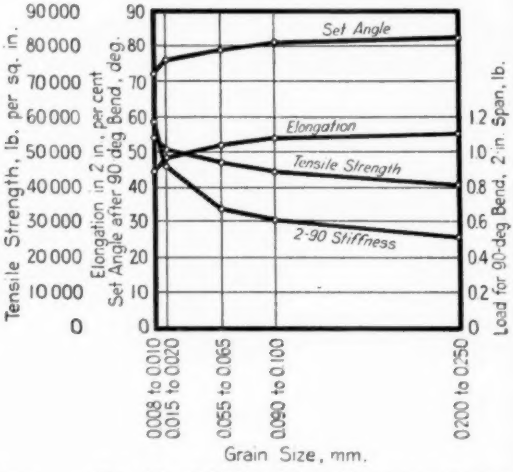


FIG. 10.—0.027 by $\frac{3}{8}$-in. 70 per cent Copper, 30 per cent Zinc Brass Strip.

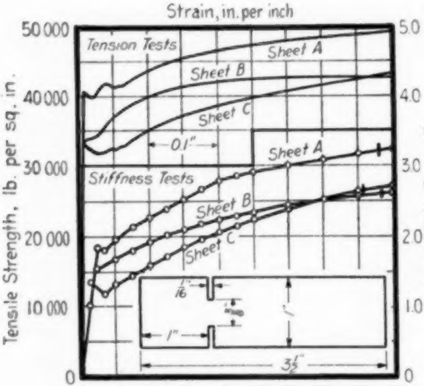


FIG. 11.—No. 20-Gage Auto-Body Sheet.

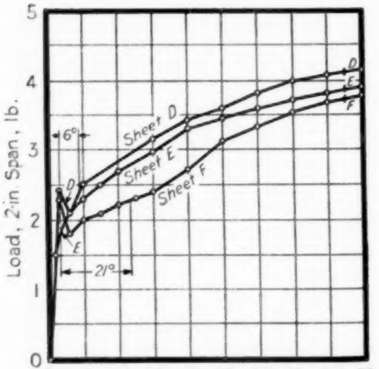


FIG. 12.—No. 20-Gage Auto-Body Sheet.

with elongation and "2–90 stiffness" with tensile strength. Specimens were not notched.

Hot-rolled or annealed steel sheet gives a stiffness test curve somewhat similar to that of the tension test. The extremely rapid yield and elongation at this point in the tension test are paralleled closely in the stiffness test. In the tests made in Fig. 11, narrowly-notched specimens were used in the stiffness tests. Load was applied with the motor, at constant speed, until yield. At this point the machine was stopped and the specimen permitted to settle, deforming quickly under the weight of the pendulum. The motor was then thrown in gear and the test completed. This method was adopted because of the difficulty in observing and recording stress and strain data where the yield took place quickly.

In Fig. 11 stiffness tests are plotted on three specimens of No. 20 gage auto body sheet. Specimens were notched according to the outline shown. Sheet *B* is a skin-passed sheet. Yield point deformation is low. Sheets *A* and *C* are not skin-passed and both show sudden yield and considerable yield point deformation. Relative spring-back of these sheets is shown by the "set angle" readings, the difference between set angle and 90 deg. being the index. All three sheets were 0.0375 in. thick. Bends were made across grain. Sheets *A* and *C* show greater tendency to work harden, the curve rising steeply as deformation progresses, both in the tensile and bending stress-strain diagrams. Toughness of the sheet is proportional to the area under the curve, and, as shown by both tension and stiffness tests, is greater for sheets *A* and *C*.

Figure 12 illustrates stiffness tests on three more specimens of 0.0375-in. auto body sheet. Sheets *D* and *F* are dead-soft annealed and sheet *E* has been recently roller-leveled. Sheets *D* and *F* showed many stretcher strains on drawing. The stiffness test specimens also showed these markings plainly at the points of bend. The stretcher strains on sheet *F* were much deeper and more extensive. As a comparative index of this stretcher strain tendency the measurements shown in Fig. 12, in this case 6 deg. for sheet *D* and 21 deg. for sheet *F*, are suggested, this being the deformation between the yield point and the subsequent work hardening until load again equals that at yield point. Tension tests were as follows on these samples:

	Sheet D	Sheet E	Sheet F
Tensile strength, lb. per sq. in....	42 400	43 400	42 200
Yield point, lb. per sq. in..........	30 500	25 500	28 800
Elongation in 2 in., per cent.......	40	42.5	48.5
Yield point elongation, per cent...	2.7	0.6	3.65

In the stiffness test of steel sheet most of the values pertinent may be read directly from the scales or computed from dial readings without plotting a curve. These are: the yield point in flexure (load and angle), point where load again reaches yield value, the 90-deg. stiffness load, the spring-back and the toughness factor, which is an average of the observed load readings at, say, every 10 deg.

Acknowledgment.—The author wishes to thank the organizations who furnished specimens for test and also to express his appreciation of the assistance given by members of Committee B-4 and of his own organization.

DISCUSSION

Mr. Dean Harvey.[1]—The testing of the temper of wire, strip and sheet, is a matter of a great deal of importance. I have been told that in the radio tubes alone more than half a million pounds of nickel wire were used last year. The radio tube manufacturers are much interested in obtaining a uniform temper for this wire in order to make a product of the desired quality and to expedite manufacture, so they have been working for several years to devise a satisfactory test for stiffness. At their request, Committee B-4 on Electrical-Heating, Electrical-Resistance and Electric-Furnace alloys has undertaken the development of a bend test. In the 1936 report of the committee a preliminary draft of three methods of bend testing of wire was included for information.[2] One of the three machines described is the machine which Mr. MacBride presented in his paper today. He has been an active member of Subcommittee IV on Mechanical Tests of Committee B-4, which subcommittee is working on this subject. We are trying to develop one standard machine instead of having three or four different machines giving results which are not readily compared. The tension test does not tell the whole story as regards the stiffness of the wire, and a test which will actually bend the wire, thus more nearly corresponding to actual use, is preferable, although tension tests give valuable information.

Mr. W. M. Patterson.[3]—I think Mr. MacBride has done a fine bit of laboratory work in lining up a new tool which can be used in obtaining information on materials which we have previously found difficult to test. Further work, however, seems indicated at a few points.

A recent improvement in testing sheet has been to confine the plastic deformation to a small localized section by means of a standard notch close to the vise face. If this is not done, work hardening causes the deformation to move out towards the point of loading, bringing a further factor into the test. Similar confinement should be applied to wire and strip tests. Strip can be handled by use of a small notch, and wire by additional support of the portion in which no deformation is desired.

Mr. MacBride has explained the close agreement with the flexure formulas of results throughout the entire test, by considering the material to be in a pseudo-elastic state throughout, with each stress-strain increment giving a sort of modulus of elasticity without regard to whether the deformation is elastic or plastic. Since both the load and deflection formulas

$$W = \frac{3EID}{L^3} \text{ and } D = \frac{WL^3}{3EI}$$

contain the modulus as a theoretic constant, this seems to be an incorrect analysis, particularly as a thinner sheet

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[1] Materials Engineer, Central Engineering Laboratories and Standards, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 162 (1936).

[3] Engineer of Tests, Parker-Kalon Corp., New York City.

or smaller diameter wire will take a greater angular deflection before plastic deformation starts than will a heavier one of the same material.

I believe that the correlation works well because of the relatively small change of cross-section and the continued elastic behavior of the fibers nearer the neutral axis, which have not been overstrained.

Mr. MacBride is to be congratulated on his ingenious correction chart which may have many applications in other engineering work.

I think there is room for further work to determine the effect of vise pressure in inducing stresses in addition to the applied bending stresses. Attention may also be called to the fact that looping of the specimen occurs between vise and loading pin, which will change the gage length and affect deflection readings, when a notch is not used.

For certain materials showing a marked yield point, the author recommends stopping the machine and permitting the test to reach static equilibrium before the reading is taken, rather than continuing the dynamic equilibrium supplied by the constant speed motor as used for the rest of the readings, and throughout for tests not showing a marked yield point. By having two men take simultaneous readings of load and deflection, the test would not have to be changed for this special condition, and the uniformity of test procedure would more than make up for the inconvenience.

While it is always questionable practice to bring a new test into a field in which there are already a great many, the writer feels that there are more than enough advantages in this type of stiffness test to justify its addition to the present means of checking materials.

The writer believes that in many cases the stiffness test is more sensitive to small variations than the tension test. The load readings check well with the tensile strength, and the set angles check well with elongation. The specimen is more easily machined than the standard tension test specimen. The machine requires little experience to operate.

In conclusion, I wish to compliment the author on his excellent work in indicating how the test results may be correlated with information wanted in actual manufacturing practice. The ultimate test of the machine of course will be its use in determining the acceptability of materials for commercial applications.

MR. SAM TOUR.[4]—In many jobs we are often inhibited by knowing too much. In this particular type of test we have had just that type of inhibition from the start. The standard cantilever beam formula in normal structural design is based on several assumptions: First, that the neutral axis does not shift as a result of the load applied; second, that the gage length of the specimen or of the structure does not vary due to the angle of deflection; third, that the section stressed does not change its shape; and fourth, that the modulus of elasticity is a constant. In a cantilever beam test in which a considerable amount of deflection is given to a specimen and in which there is considerable actual plastic working at the point of maximum bending, none of those assumptions holds true. Therefore, the standard cantilever beam formula cannot apply. The tendency, then, on the part of some of us who lean towards these theoretical considerations is to use the theoretical formula on a few results and say: "See, it does not work; the thing is out of line entirely with the good standard accepted theory

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[4] Vice-President, Lucius Pitkin, Inc., New York Cit

for many years, and, therefore, it is no good." The practical angle of it, however, is that we can discard those points of dissimilarity between a cantilever beam test on a specimen which is loaded beyond its elastic limit and a test where the specimen is loaded within the elastic limit—we can discard those deviations on the ground solely that the results obtained in a cantilever beam test do give us information of value in industry and do tell us things that other tests have not been able to tell us in the past, and tell us these things with a minimum of time and effort.

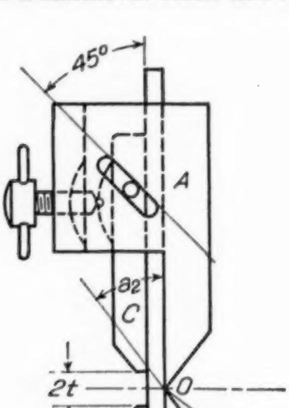

FIG. 1.—Clamp Arranged So that Bending Produces Nothing but Stretching in Test Piece.

There was a question raised regarding what could be done with round wire to take the place of the reduced section used on strip. The need for necking decreases as the gage length of the specimen tested is decreased. The tendency to use a 4-in. gage length of specimen exaggerates the need for a reduced section. A 2-in. specimen does not develop as much "free bend" within the gage length, and similarly a ½-in. gage length eliminates still more of it. By using the shortest possible gage length some of the errors due to the progress of bending from the edge of the fulcrum out to the point of loading is eliminated.

It might be of interest to point out one or two other types of tests which can be carried out on the machine which Mr. MacBride has described. By supporting a specimen in the grip in a vertical position, lifting the loading pin to any point of loading desired and suddenly releasing it, the impact bending type of test can be carried out very readily on this machine. By holding the specimen in the horizontal position, but bringing the loading lever up to the load position and adjusting the pin against the specimen and then releasing the loading lever, the "force-bending" type of test can be carried out. In other words, this one machine may be used to carry out the three types of test which Mr. Harvey spoke of in connection with the work of Committee B-4.

MR. E. E. THUM.[5]—It is rather interesting that bend testing, which is a very early type of metallurgical test, is now getting more and more attention in an effort to gage the results of the old qualitative bending test in such a way that it can become quantitative.

I have observed a somewhat different machine than the one described in some use by consumers of tin plate. The methods of testing are much the same as this, but the straight strip is gripped in vises as shown by the accompanying Fig. 1 so arranged that the pointed lower jaw of the two vises meet at the center

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[5] Editor, *Metal Progress*, American Society for Metals, Cleveland, Ohio.

of rotation of the bending device. Rotation of the disk holding the upper vise will—or, at least, is said to—put nothing but tension into the test strip, and this is supposed to be one of the advantages of this particular type of gripping. The machine in operation is capable of bending the strip to a 90-deg. angle, straightening the strip back again, and then repeating the bend, plotting one curve after another until fracture ensues. The number of bends of that sort which the material will stand, and the relation between the various load-bend curves automatically drawn, is supposed to be indicative of quality.

I believe there are a few of such foreign-built machines now in operation in America. From what I have heard, the users of tin plate are much more anxious to use such a machine than are the producers of tin plate.

I should also like to comment on Fig. 2 of the paper. This correction chart for various thicknesses would probably be beyond criticism for corrections of a relatively few per cent, but one would need to scrutinize the results if results were to be corrected by, say, 50 per cent or more.

MR. H. L. MACBRIDE[6] (*author's closure, by letter*).—Replying to Mr. Patterson and Mr. Thum, the cube-of-thickness correction for sheet metal is sufficiently accurate when the variation from gage is not more than the usually encountered 5 or 10 per cent.

Mr. Patterson is no doubt correct in saying that plastic deformation will take place at smaller angular deflections in thicker specimens. However, with thickness variations of 5 or 10 per cent, the observed proportional limit in bending seems to take place within a small difference of angular deflection.

The deflection formulas shown for cantilever beams merely called attention to the fact that in all bending formulas, the load, in theory, varies directly with the moment of inertia of the cross-section. If the width remains constant, load should vary as the cube of thickness. The modulus of elasticity is merely a ratio between load and deformation, and for any small increments of load might be called modulus of deformation. This ratio is constant during the beginning of the progressive deflection, and then at some point in the continuous bending becomes a variable, its rate of change depending upon the nature of the material and, of course, to some extent upon the shape of the specimen. While it is true that the outer fibers are stressed to their elastic limit before those nearer the neutral axis, it seems to me safe to assume that where the difference in thickness between two specimens is small, the deflection in the progressive bend of each specimen where this elastic failure occurs should be about the same, provided the specimens are identical in structure. Pyroxylin sheets, that is, materials wholly plastic, have been sanded to slightly different thicknesses and the cube-of-thickness correction throughout the bend found to check closely.

Mr. Tour said that the neutral axis must be presumed not to shift during the bending. This is of course true in applying the beam formulas to elastic deflections. However, I do not agree with this under the special conditions which I have assumed, because the load is, in theory, proportional to the cube of thickness, regardless of the distance of the neutral axis from the top or bottom of the specimen. I only assume that if the neutral axis does shift, because of change in the ratio of deformation in compression and tension, the rate of shift will be practically the same in two specimens of identical structure where the difference in thickness is relatively small.

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[6] Research Engineer, Tinius Olsen Testing Machine Co., Philadelphia, Pa.

FATIGUE PROPERTIES OF NON-FERROUS SHEET METALS

By C. H. Greenall[1] and G. R. Gohn[1]

Synopsis

This paper continues an investigation described in a previous paper before the Society.[2] Results are given of fatigue tests on nine alpha-brass alloys, one nickel-silver alloy, two phosphor-bronze alloys, three beryllium-copper alloys, one copper-nickel-silicide alloy, copper, Everdur, nickel, three nickel alloys and three aluminum alloys. The effect of grain direction on the fatigue properties of nickel silver and three aluminum alloys is shown. The effect of surface treatment and electroplated finishes on the fatigue properties of phosphor bronze is discussed as is the effect of grain size and surface treatment on the fatigue properties of nickel silver sheet.

The data show that nickel silver sheet of 0.015 to 0.017-mm. average grain size has a higher endurance limit than similar material of larger grain size.

The data also confirm the conclusions of the previous paper,[2] that dispersion hardening of alpha brass by the addition of nickel silicide increases the endurance limit and that cold work raises the endurance limit but not proportionally to the increase in tensile strength.

Age hardening markedly increases the endurance limit of type K monel metal, aluminum alloy 17S and beryllium-copper alloys.

The ratio of endurance limit to tensile strength for the alloys investigated varies from 0.136 to 0.403, depending upon composition, heat treatment and amount of cold work. The highest endurance limits were observed for nickel and alloys of high nickel content. Little difference was observed between the endurance limits for heat-treated copper-base alloys such as beryllium copper and cold-worked alloy C phosphor bronze.

Most of the materials referred to in this paper have been discussed in three previous papers[2,3,4] covering the development of specification requirements for non-ferrous materials and the determination of their fatigue properties. These materials are widely used in the manufacture of a variety of electromechanical devices in which they are frequently employed as electrical contact springs. Under normal service conditions, those springs are subjected to millions of cycles of stress; hence it is essential that design data be available which will permit the choice of a material which will not fail due to fatigue under operating stresses.

The design and operation of the machines developed for obtaining these fatigue data have been previously de-

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[1] Members of Technical Staff, Bell Telephone Laboratories, Inc., New York City.

[2] J. R. Townsend and C. H. Greenall, "Fatigue Studies of Non-Ferrous Sheet Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part II, p. 353 (1929).

[3] H. N. Van Deusen, L. I. Shaw and C. H. Davis, "Physical Properties and Methods of Test for Sheet Brass," *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part II, p. 173 (1927).

[4] J. R. Townsend, W. A. Straw and C. H. Davis, "Physical Properties and Methods of Test for Some Sheet Non-Ferrous Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part II, p. 381 (1929).

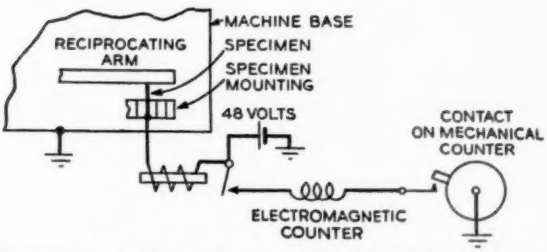


FIG. 1.—Schematic Drawing of Monitoring Circuit for Sheet Metal Fatigue Testing Machine.

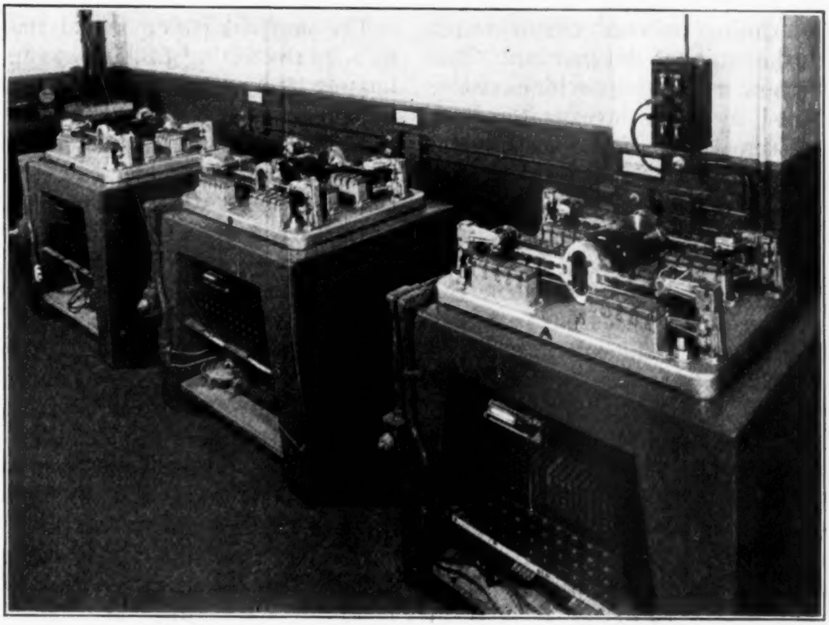
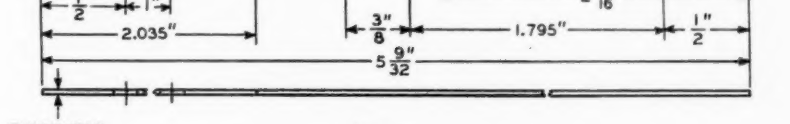


FIG. 2.—Sheet Metal Fatigue Testing Machines.



PIECE MUST BE FREE FROM SCRIBER AND TOOL MARKS

— NOTE —
THE 2-INCH RADIUS FILLETS MUST JOIN THE TAPERED PORTION WITHOUT AN ABRUPT BREAK. THE INTERSECTION OF THE TAPERED SIDES EXTENDED MUST BE ½-INCH FROM THE END.

RATIO $\frac{l}{b} = 9.287$

FIG. 3.—Test Specimen.

scribed.[2] The same machines have been used unchanged in the present investigation except for the addition of a monitoring circuit (Fig. 1) which is used for recording individual failures of the fatigue specimens. During test, each specimen is connected into the monitoring circuit as shown in Fig. 1. Failure of the specimen causes the relay to release, closing another circuit which starts the counter mechanism. This machine and monitoring circuit have also been used by other investigators.[5] A view of three units of the sheet metal fatigue testing machine is shown in Fig. 2.

The test specimen shown in Fig. 3 has been changed slightly from that described in the previous paper.[2] Experience during the past eight years has indicated that it is difficult to obtain a satisfactory form milling cutter from the method of dimensioning previously used. Accordingly, another and more complete method of dimensioning the test specimen is shown. The basic dimensions are now referred back to the $\frac{15}{32}$-in. end, from which end all specimens are located in the testing machine. This change has necessitated an increase in the 2-in. dimension previously used to a value of 2.035 in. The location of the uniformly stressed area, however, remains the same with respect to the intersection of the tapered sides projected, that is, the point of actuation of the specimen under test. A more thorough mathematical analysis of the shape of the specimen has shown that the ratio of length to thickness, l/b, should be increased from 9.269 to 9.287.

The specimens for test are prepared in a manner similar to that described elsewhere for the preparation of tension test specimens.[6] The material is cross-milled with a straight-fluted, form milling cutter, extreme care being employed to prevent the formation of transverse grooves during the milling operation which might affect the fatigue results. All specimens were cut with the direction of rolling as indicated in Table II.

The previous paper[2] stated that the speed of the testing machine was approximately 1500 r.p.m. It was pointed out, however, that it was necessary to adjust the speed of the machine, depending upon the material under test, in order that the speed did not approach the natural frequency of the test specimen. The majority of the tests previously reported were made at a testing speed of 750 to 900 r.p.m. Present tests, with the exception of the test data also reported in the previous paper, were made at 750 r.p.m. This is comparable to the speed used by Price and Bailey.[5] Each sheet metal fatigue machine has a capacity of 40 specimens, 20 specimens being tested at a given deflection. Previous tests were made on 5 specimens for each deflection, but because of the number of materials studied, many of the tests reported in this investigation were limited to three specimens at each deflection.

The deflection of the reciprocating arm is first adjusted while the machine is stationary. The final adjustment is made while the machine is running at the testing speed. This is accomplished by having a small angle bracket attached to each reciprocating arm. Electrical contact is made with this angle at both ends of the stroke and the dynamic deflection is readily obtained by measuring the distance between two electrical

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[5] W. B. Price and R. W. Bailey, "Fatigue Properties of Five Cold Rolled Copper Alloys," *Technical Publication No. 786*, Am. Inst. Mining and Metallurgical Engrs., New York Meeting, February, 1937.

[6] R. L. Templin "Methods for Determining the Tensile Properties of Thin Sheet Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part II, p. 235 (1927).

contacts. It is felt that this method is more accurate and simpler to use than the one previously employed using a stroboscope.

The sheet materials investigated consist of nine alpha-brass alloys, one nickel-silver alloy, two phosphor-bronze alloys, three beryllium-copper alloys, one copper-nickel-silicide alloy, copper, Everdur, nickel, three nickel alloys and three aluminum alloys. These materials had been cold worked varying amounts prior to testing. In hardness, the materials varied from annealed to spring and extra spring tempers. Certain of the alloys were also heat treated or subjected to special surface treatments prior to testing.

The average chemical composition for the materials tested is given in Table I. Table II lists the physical properties of the materials studied as well as heat treatment, special surface preparation and the minimum fatigue limit for each material at 100,000,000 cycles of completely reversed stress as determined from *S-N* curves plotted from the test data tabulated in Table III.

The method for determining the stress in the fatigue test specimen has been described in the previous paper.[2]

Certain of the test results illustrating the average *S-N* relationship are shown in Figs. 4 to 9, which are plotted from the data in Table III. To avoid confusion the individual points are not plotted. The data of Table III will enable those interested to plot the *S-N* curves and to check the endurance limits of all materials reported in Table II.

Micrographic examination of broken test specimens of various materials indicate the type of failure to be similar to that previously shown in Fig. 11 of the earlier paper.[2] The fatigue cracks have been without exception transcrystalline and no distortion of the metal adjacent to the fracture has been observed.

A study of the test data confirms the conclusion of the previous paper[2] that the ratio of endurance limit to tensile strength for non-ferrous sheet metals is much lower than that reported for steel. The ratio of endurance limit to tensile strength for the materials studied varied from 0.136 to 0.403, which is approximately half of the ratio reported for heat-treated steel.[7]

Examination of the data shown in Table II indicate that, in general, the ratio of endurance limit to tensile strength decreases with increasing amounts of cold work. Attention is called, however, to the data for alloy A phosphor bronze and monel metal, in which case the ratio of endurance limit to tensile strength increased for intermediate amounts of cold working.

Whereas the earlier paper[2] had reported, on the basis of a limited number of tests, the endurance limits presented in the third column of the following table, more complete tests indicate that these values should be those listed in the fourth column:

MATERIAL	B. & S. NUMBERS HARD	ENDURANCE LIMIT PREVIOUSLY REPORTED, LB. PER SQ. IN.	ENDURANCE LIMIT, LB. PER SQ. IN.
Alloy A brass......	0	12 000	15 000
	4	13 500	19 500
	10	15 000	20 000
Alloy G brass......	0	12 000	13 000
	4	18 000	20 000
Alloy B nickel silver............	0	14 000	17 500
	4	18 500	21 500
	10	22 000	21 500
Everdur...........	8	24 000	23 500
Alloy No. 33.......	..	14 000	18 500
No. 34.......	..	12 500	16 000
No. 35.......	..	16 000	20 500

Likewise it has been found that the *S-N* curves for the non-ferrous metals do not exhibit the sharp knees characteristic of similar curves for steel. Hence

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[7] H. J. Gough, "The Fatigue of Metals," Scott, Greenwood and Sons; also H. F. Moore and J. B. Kommers, "The Fatigue of Metals," McGraw-Hill Book Co., Inc., New York City (1927).

TABLE I.—AVERAGE CHEMICAL ANALYSIS FOR NON-FERROUS SHEET MATERIALS TESTED.

Alloy	Copper, per cent	Lead, per cent	Iron, per cent	Zinc, per cent	Tin, per cent	Nickel, per cent	Other Elements, per cent	Other Impurities, per cent
High brass								
Rolling series	65.09	0.02	0.03	Remainder	0.00			
Commercial	64.78	0.04	0.02	Remainder				
Hot-rolled rolling series	65.01	0.12	0.06	Remainder	0.00			
Alloy G brass								
Rolling series	71.73	0.02	0.03	Remainder	0.00	0.01		
Commercial	71.68	0.01	0.02	Remainder				
Alloy B nickel, silver								
Rolling series	55.23	0.005	0.06	Remainder		18.38	0.11 manganese	0.018 carbon
Commercial (first lot)	55.09		0.04	26.31		18.35	0.16 manganese	
Commercial (second lot)	54.99	0.01	0.04	26.30		18.55	0.11 manganese	
Special	55.18	Trace	0.03	27.08		17.60	0.14 manganese	
Commercial[a] (third lot)	53.50 to 56.50	0.10 max.	0.35 max.	25.50 to 28.50		16.50 to 19.50	0.50 max. manganese	
Copper								
Commercial	99.95							
Everdur								
Rolling series	95.46						0.99 manganese 3.55 silicon (by difference)	
Commercial[a] (first lot)	96.00						1.00 manganese, 3.00 silicon	
Commercial (second lot)	95.89		0.11	0.14		0.08	0.74 manganese, 3.02 silicon	
Alloy A phosphor bronze								
Commercial	95.82	Nil	0.04	0.09	3.86		0.068 phosphorus	0.00 antimony
Alloy C phosphor bronze								
Rolling series	91.84	0.02	0.03	0.00	8.08	0.00	0.03 phosphorus	
Commercial	92.04	0.01	0.02	0.16	7.68		0.066 phosphorus	0.00 antimony
Commercial[a] (finish studies)	91.0 min.	0.02 max.	0.10 max.	0.20 max.	7.50 to 8.50		0.05 to 0.35 phosphorus, 0.01 max. antimony	Trace
Beryllium-copper								
Lot P[a]	Remainder						2.25 beryllium	
Lot OP[a]	97.44					0.31	2.15 beryllium	0.06 iron, 0.03 silicon
Lot 2P[a]	97.50					0.32	2.16 beryllium	0.06 iron, 0.03 silicon
Lot 4P[a]	97.54					0.28	2.14 beryllium	0.06 iron, 0.03 silicon
Lot 5[a]	Remainder					0.50	2.25 beryllium	
Lot 6[a]	Remainder					0.50	2.25 beryllium	
Lot 7[a]	Remainder					0.50	2.25 beryllium	
Dispersion hardened copper								
Alloy No. 80	94.15					5.14	0.71 silicon (by difference)	
Dispersion hardened brasses								
Alloy No. 33	Remainder			9.89		2.32	0.57 silicon	
Alloy No. 34	Remainder			19.89		2.37	0.57 silicon	

Alloy No. 35	Remainder	...	...	30.12	...	2.36	0.66 silicon	...
Alloy No. 79	67.18	...	...	30.14	...	2.45	0.63 silicon	...
Alloy No. 85	69.80	...	...	27.20	...	2.11	0.54 silicon	...
Alloy No. 77	69.98	...	...	27.39	...	2.33	0.63 silicon	...
Alloy No. 78	68.27	...	...	27.39	...	3.90	0.98 silicon	...
Monel metal								
Rolling series	28.26	...	1.47	...	...	68.65	...	1.16 manganese, 0.10 carbon
Commercial	29.82	...	1.62	...	...	67.43	...	1.06 maganese, 0.26 sulphur
Nickel	0.09	...	0.22	...	...	Remainder	0.48 cobalt	0.14 manganese, 0.14 silicon
High silicon monel metal	29.37	...	0.55	...	...	68.31	1.85 silicon	...
Type K monel metal	29.24	...	1.57	...	...	64.35	0.38 manganese, 3.80 aluminum	0.001 silicon, 0.18 carbon
Aluminum alloy 2S	0.12	...	0.52	...	...	...	99.21 aluminum (by difference)	Trace manganese, 0.15 silicon, 0.00 magnesium
Aluminum alloy 3S	0.11	...	0.50	...	...	...	1.16 magnesium, 98.04 aluminum (by difference)	0.195 silicon, trace magnesium
Aluminum alloy 17S[b]	3.15–4.50	...	0.50 max.	...	...	...	0.40 to 1.00 magnesium, 0.40 to 1.00 manganese, Remainder aluminum	0.03 max. including zinc

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[a] Nominal composition.

[b] Proposed composition limits.

TABLE II.—PHYSICAL PROPERTIES.

Material	B. & S. Gage	B. & S. Numbers Hard	Condition or Heat Treatment	Grain Size Ready-to-Finish Anneal, mm.	Direction of Rolling with Respect to Length of Specimen	Tensile Strength, lb. per sq. in.	Proportional Limit, lb. per sq. in.	Modulus of Elasticity, lb. per sq. in.	Elongation in 2 in., per cent	Rockwell Hardness, "B" scale	Endurance Limit at 10⁸ Reversals of Stress, lb. per sq. in.	Ratio, endurance limit to tensile strength
High brass Rolling series	24	0	600 C. anneal	...	Parallel	46 600	13 000	14 500 000	58	16	15 000	0.322
		4	...	...	Parallel	77 200	32 000	...	6	79	19 500	0.253
		10	...	...	Parallel	95 600	30 000	...	2	87	20 000	0.209
Commercial	24	0	...	...	Parallel	46 600	9 500	14 840 000	57	14.2	13 500	0.290
		4	...	...	Parallel	73 700	31 900	15 620 000	6.5	79.1	17 500	0.237
		8	...	...	Parallel	91 900	33 100	14 970 000	1.5	87.3	17 500	0.190
Hot rolled rolling series	24	0	...	...	Parallel	...	...	...	...	...	13 500	...
		4	...	...	Parallel	69 600	36 200	...	4.8	73	15 000	0.216
		10	...	...	Parallel	93 900	36 100	...	2.4	89	20 500	0.218
Alloy G brass Rolling series	24	0	600 C. anneal	...	Parallel	46 300	...	...	61	16	13 000	0.281
		4	...	...	Parallel	81 600	...	...	6	84	20 000	0.245
		10	...	...	Parallel	97 800	...	...	2	92	20 000	0.204
Commercial	24	0	...	...	Parallel	49 400	11 800	15 600 000	54.0	23.5	16 500	0.334
		4	...	...	Parallel	75 200	23 100	15 860 000	7.5	81.7	19 000	0.253
		8	...	...	Parallel	93 800	36 700	14 860 000	1.0	90.1	22 000	0.235
Alloy B nickel silver Rolling series	24	0	...	...	Parallel	66 900	...	20 000 000	42	19[a]	17 500	0.262
		4	...	...	Parallel	98 700	...	...	2	69[a]	21 500	0.218
		10	...	...	Parallel	116 200	...	...	1.5	79[a]	21 500	0.185
Commercial	24	0	...	...	Parallel	62 200	12 300	18 060 000	45.5	...	16 000	0.257
		6	...	...	Parallel	102 200	48 300	20 320 000	1.2	...	21 000	0.205
		8	...	...	Parallel	113 400	50 000	19 470 000	1.0	...	22 500	0.198
Commercial	24	0	...	...	Parallel	60 500	...	...	48	53	16 500	0.273
			...	...	45 deg.	62 800	15 400	14 700 000	49	53	17 500	0.279
			...	...	90 deg.	60 400	...	...	48	53	14 000	0.232
		4	...	...	Parallel	95 400	...	...	1.5	66.1[a]	20 500	0.215
			...	...	45 deg.	94 400	44 000	18 000 000	2.4	66.1[a]	22 000	0.233
			...	...	90 deg.	93 200	...	...	5.3	66.1[a]	18 500	0.198
		10	...	...	Parallel	117 700	...	...	1.2	78.6[a]	25 000	0.212
			...	...	45 deg.	114 800	48 900	16 700 000	1.5	78.6[a]	26 500	0.231
			...	...	90 deg.	124 600	...	...	2.0	78.6[a]	28 000	0.225

Special	24	6	......	0.017	Parallel	106 600	......	......	......	......	25 500	0.239
		6	Annealed 1/2 hr. at 330 C.	0.017	Parallel	110 000	......	......	......	......	26 500	0.241
		6	......	0.022	Parallel	108 500	......	......	......	......	23 000	0.212
		6	Annealed 1/2 hr. at 330 C.	0.022	Parallel	114 300	......	......	......	......	24 000	0.210
		6	......	0.090	Parallel	108 100	......	......	......	......	23 000	0.213
		6	Annealed 1/2 hr. at 330 C.	0.090	Parallel	110 900	......	......	......	......	22 500	0.203
	24	8	......	0.015	Parallel	114 200	......	......	......	......	28 250	0.247
		8	Annealed 1/2 hr. at 330 C.	0.015	Parallel	118 500	......	......	......	......	28 750	0.243
		8	......	0.020	Parallel	117 200	......	......	......	......	26 500	0.226
		8	Annealed 1/2 hr. at 330 C.	0.020	Parallel	125 400	......	......	......	......	29 500	0.235
		8	......	0.090	Parallel	112 400	......	......	......	......	23 750	0.211
		8	Annealed 1/2 hr. at 330 C.	0.090	Parallel	118 250	......	......	......	......	24 500	0.207
Commercial	24	6	Control specimens	......	Parallel	102 200	48 300	20 320 000	1.2	......	21 000	0.205
		6	1/32 by 1/32-in. square hole	......	Parallel	102 200		......	......	......	20 250	0.198[d]
		6	1/32 by 3/64-in. rect. hole	......	Parallel	102 200		......	......	......	15 500	0.152[d]
		6	1/32 by 1/16-in. rect. hole	......	Parallel	102 200		......	......	......	17 900	0.175[d]
		6	0.001-in. scratch—one side	......	Parallel	102 200		......	......	......	21 600	0.211[d]
		6	90 deg. bend—3-1/32-in. radii	......	Parallel	102 200		......	......	......	17 000	0.166[d]
		6	1/8-in. offset—1/32-in. radii	......	Parallel	102 200		......	......	......	17 000	0.166[d]
Copper												
Commercial	24	0	......	......	Parallel	31 400	6 800	......	35.0	......	11 000	0.350
		2	......	......	Parallel	44 400	15 900	......	7.8	33.2	13 000	0.293
		6	......	......	Parallel	52 600	14 700	......	2.2	55.3	14 000	0.266
Everdur												
Rolling series	24	8	......	......	Parallel	80 000	26 000	12 400 000	22	91	23 500	0.294
Commercial, first lot	24	8	......	......	Parallel	......	......	......	......	......	25 000	......
Commercial, second lot	24	0	......	......	Parallel	59 300	9 400	16 600 000	63	41.4	16 000	0.270
		4	......	......	Parallel	93 700	28 500	15 610 000	7.2	91.0	23 000	0.245
		8	......	......	Parallel	107 600	31 600	15 370 000	2.0	95.3	20 500	0.191
Alloy A phosphor bronze												
Commercial	24	0	......	......	Parallel	55 100	13 300	12 800 000	52	13.9	13 750	0.250
		4	......	......	Parallel	78 800	52 500	15 500 000	6.3	53.9[a]	25 500	0.324
		8	......	......	Parallel	90 200	54 400	14 800 000	1.8	56.6[a]	22 000	0.244
Alloy C phosphor bronze												
Rolling series	24	0	......	......	Parallel	59 700	26 000	15 490 000	67	11[a]	21 000	0.352
		4	......	......	Parallel	95 500	......	......	14	71[a]	22 000	0.230
		10	......	......	Parallel	124 800	55 000	15 000 000	2	84[a]	24 500	0.196
Commercial	24	0	......	......	Parallel	51 800	12 800	15 490 000	78	33.8	17 250	0.333
		4	......	......	Parallel	88 000	52 100	15 700 000	18.5	64.7[a]	22 000	0.250
		8	......	......	Parallel	112 900	56 100	15 060 000	1.5	79.5[a]	27 000	0.239

TABLE II.—PHYSICAL PROPERTIES—*Concluded.*

Material	B. & S. Gage	B. & S. Numbers Hard	Condition or Heat Treatment	Grain Size Ready-to-Finish Anneal, mm.	Direction of Rolling with Respect to Length of Specimen	Tensile Strength, lb. per sq. in.	Proportional Limit, lb. per sq. in.	Modulus of Elasticity, lb. per sq. in.	Elongation in 2 in., per cent	Rockwell Hardness, "B" scale	Endurance Limit at 10^8 Reversals of Stress, lb. per sq. in.	Ratio, endurance limit to tensile strength
Alloy C phosphor bronze—*Contin'd* Commercial	24	8	Unfinished control specimens	......	Parallel	115 300	58 600	15 530 000	2.0	81.2[a]	26 000	0.226
		8	Finish A	......	Parallel	115 300	....	....	......	....	24 000	0.208[d]
		8	Finish B	......	Parallel	115 300	....	....	......	....	23 500	0.204[d]
		8	Finish C	......	Parallel	115 300	....	....	......	....	19 000	0.165[d]
		8	Finish D	......	Parallel	115 300	....	....	......	....	27 000	0.235[d]
		8	Finish E	......	Parallel	115 300	....	....	......	....	26 000	0.226[d]
		8	Finish F	......	Parallel	115 300	....	....	......	....	26 000	0.226[d]
		8	Microscopic polish	......	Parallel	115 300	....	....	......	....	28 500	0.247[d]
Beryllium-copper												
Lot P	18	0	Annealed at 800 C. Quenched in water. Aged 2 hours at 275 C.	......	Parallel	171 200	....	19 100 000	4.8	110.9	31 000	0.181
Lot OP	18	0	Annealed at 800 C. Quenched in water. Aged 2 hrs. at 275 C.	......	Parallel	175 000[c]	46 000[c]	18 900 000[c]	6.3[c]	102[a,c]	28 000	0.160
Lot 2P	18	2	Annealed at 800 C. Quenched in water. Cold rolled. Aged 2 hrs. at 275 C.	......	Parallel	173 000	48 000[c]	18 500 000[c]	4.8[c]	102[a,c]	27 250	0.158
Lot 4P	18	4	Annealed at 800 C. Quenched in water. Cold rolled. Aged 2 hrs. at 275 C.	......	Parallel	193 000[c]	55 000[c]	18 400 000[c]	2.0[c]	104.5[a,c]	28 000	0.145
Lot 5	24	4	Annealed at 800 C. Quenched in water. Cold rolled	......	Parallel	118 000[c]	39 000[c]	17 200 000[c]	4.3[c]	84[a,c]	27 000	0.229
Lot 5—aged	24	4	Aged 2 hours at 275 C. after cold rolling	......	Parallel	193 000[c]	55 000[c]	18 400 000[c]	2.0[c]	104.5[a,c]	36 000	0.187
Lot 6	18	2	Annealed at 800 C. Quenched in water. Cold rolled	......	Parallel	103 000[c]	33 000[c]	17 500 000[c]	8.0[c]	79[a,c]	23 250	0.226
Lot 6—aged	18	2	Aged 2 hrs. at 275 C. after cold rolling	......	Parallel	173 000[c]	48 000[c]	18 500 000[c]	4.8[c]	102[a,c]	25 500	0.147
Lot 7	24	2	Annealed at 800 C. Quenched in water. Cold rolled	......	Parallel	84 100	25 900	19 400 000	19.1	85.9	23 000	0.274
Lot 7—aged	24	2	Aged 2 hrs. at 275 C.	......	Parallel	171 000	49 500	19 800 000	4.0	107.6	26 500	0.155
Lot 7—aged	24	2	Aged 1 hr. at 275 C.	......	Parallel	....	....	....	......	....	30 000	......
Dispersion hardened copper	24	0	....	......	Parallel	102 600	53 200	16 400 000	4.0	....	14 000	0.136
Alloy No. 80	24	10	....	......	Parallel	133 400	38 600	15 900 000	1.0	....	24 000	0.180
Dispersion hardened brass												
Alloy No. 33	24	....	Quenched from 800 C. Aged 1 hr. at 500 C.	......	Parallel	90 000	44 500	19 800 000	14	86	18 500	0.206
Alloy No. 34	24	....	Quenched from 800 C. Aged 1 hr. at 500 C.	......	Parallel	85 800	37 200	17 200 000	21.5	85	16 000	0.186
Alloy No. 35	24	....	Quenched from 800 C. Aged 1 hr. at 500 C.	......	Parallel	85 400	38 000	16 500 000	28.0	79	20 500	0.240
Alloy No. 79	24	10	Heated 1 hr. at 800 C. Quenched in water. Aged 2 hrs. at 400 C.	......	Parallel	114 700	40 400	13 800 000	1.2	79.7[a]	18 500	0.161
Alloy No. 85	24	0	Heated 1 hr. at 800 C. Quenched in water. Aged 2 hrs. at 400 C.	......	Parallel	81 000	34 800	14 100 000	25.8	....	16 500	0.204
Alloy No. 77	24	4	Heated 1 hr. at 800 C. Quenched in water. Aged 2 hrs. at 400 C.	......	Parallel	110 800	31 100	14 000 000	2.0	77.0[a]	20 000	0.181
		10	Heated 1 hr. at 800 C. Quenched in water. Aged 2 hrs. at 400 C.	......	Parallel	114 400	32 700	13 200 000	1.6	78.9[a]	22 000	0.192

[illegible]	[illegible]	0	Heated 1 hr. at 800 C. Quenched in water. Aged 2 hrs. at 400 C.	......	Parallel	80 000	18 300	20 700 000	18.1	54.3[a]	24 000	0.300
		4	Heated 1 hr. at 800 C. Quenched in water. Aged 2 hrs. at 400 C.	......	Parallel	115 700	28 300	15 400 000	2.2	78.7[a]	21 000	0.182
		10	Heated 1 hr. at 800 C. Quenched in water. Aged 2 hrs. at 400 C.	......	Parallel	125 800	23 200	15 100 000	1.3	82.6[a]	25 500	0.203
Nickel Commercial	24	0	....	......	Parallel 45 deg. 90 deg.	72 000	12 000	22 600 000	40.4	55.4	25 000[e] 26 500[e] 23 500[e]	0.347
		4	....	......	Parallel 45 deg. 90 deg.	113 500	51 500	27 000 000	2.3	98.1	39 500[e] 40 000[e] 41 000[e]	0.352
		8	....	......	Parallel 45 deg. 90 deg.	121 500	52 000	30 300 000	2.0	100.4	40 500[e] 38 000[e] 42 000[e]	0.333
Monel metal Rolling series	22	0	....	......	Parallel	....	....	....	......	....	22 500	....
		Half Hard	....	......	Parallel	112 400	....	....	......	....	24 000	0.214
		Full Hard	....	......	Parallel	119 400	48 500	29 500 000	2.5	84.4[a]	37 500	0.314
Commercial	24	0	....	......	Parallel	73 700	21 400	23 840 000	40.5	24.9[a]	24 500	0.332
		5	....	......	Parallel	115 900	45 600	27 320 000	3.0	78.9[a]	39 500	0.341
		7	....	......	Parallel	122 800	58 200	26 420 000	1.7	82.0	37 750	0.306
High silicon monel metal	25	....	....	......	Parallel	143 500	48 500	29 300 000	2.7	90.4[a]	37 000	9.258
Type K monel metal	24	0	....	......	Parallel	88 300	....	....	38	....	26 750	0.303
	24	....	Heat treated	......	Parallel	153 000	74 000	27 700 000	11	102.4[a]	37 000	0.242
Aluminum alloys 2S	24	....	H	......	Parallel	25 800	12 400	9 100 000	3.3		9 500	0.368
			H	......	45 deg.	....	....	....	......	....	9 000	....
			H	......	90 deg.	....	....	....	......	....	9 000	....
3S	24	....	H	......	Parallel	27 800	12 400	10 000 000	5.0	....	10 000	0.360
			H	......	45 deg.	....	....	....	......	....	10 500	....
			H	......	90 deg.	....	....	....	......	....	10 500	....
17S	24	....	0	......	Parallel	31 300	10 800	9 800 000	11.5	59.9[b]	12 000	0.383
		....	0	......	45 deg.	....	....	....	......	59.9[b]	12 000	....
		....	0	......	90 deg.	....	....	....	......	59.9[b]	12 000	....
17S	24	....	T	......	Parallel	63 200	35 300	9 000 000	15.7	95.2[b]	16 500	0.268
		....	T	......	45 deg.	59 200	....	....	19.8	....	16 500	0.279
		....	T	......	90 deg.	59 800	....	....	17.4	....	16 250	0.272
17S	24	....	H	......	Parallel	38 200	21 300	9 700 000	2.5	78.8[b]	15 000	0.393
		....	H	......	45 deg.	37 200	....	....	2.2	78.8[b]	15 000	0.403
		....	H	......	90 deg.	37 800	....	....	2.5	78.8[b]	13 750	0.364

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[a] "G" scale.
[b] $\frac{1}{8}$-in. ball, 100-kg. load, red figures.
[c] Manufacturer's published data, average value.
[d] Ratio based on tensile strength of control specimens.
[e] Estimated on basis of tests being conducted.

Finish A—surface cleaned but otherwise unprepared, 10 mg. per sq. in. of nickel.
Finish B—surface cleaned but otherwise unprepared, 20 mg. per sq. in. of nickel plus flash of chromium.
Finish C—surface cleaned but otherwise unprepared, 10 mg. per sq. in. of chromium.
Finish D—surface sandblasted, 10 mg. per sq. in. of chromium.
Finish E—surface polished and sandblasted, 10 mg. per sq. in. of chromium.
Finish F—surface polished and lightly buffed, color buffed after plating, 10 mg. per sq. in. of chromium.

TABLE III—RESULTS OF FATIGUE TESTS ON VARIOUS NON-FERROUS ALLOYS.

Material	B.&S. Gage	B.&S. Numbers Hard	Direction of Rolling with Respect to Length of Specimen	Unit Stress for 1-in. Deflection, lb. per sq. in.	Number of Cycles of Reversed Stress in Thousands at Deflection in Inches Indicated in Parentheses									
High brass Rolling series.......	24	0	Parallel	50 300	(0.250)	(0.290)	(0.300)	(0.335)	(0.350)	(0.400)	(0.500)	(0.550)	(0.595)	(0.650)
					100 000→	20 189	4316	4251	2033	1648	637	224	280	123
					100 000→	100 000→	6260	6254	2248	2328	675	236	287	242
					100 000→		13 933	6874	2470	2708	675	253	295	242
					100 000→		15 944	13 488	3110			342		363
					100 000→		62 532	29 277	3684			463		363
	24	4	Parallel	49 600	(0.350)	(0.375)	(0.400)	(0.435)	(0.500)	(0.550)	(0.595)	(0.865)		
					100 000→	83 816	3191	1951	2200	973	1049	231		
					100 000→	90 638	20 239	2590	3248	1074	1150	312		
					100 000→	100 000→	22 103	2746	3248	1485	1160			
						100 000→	28 943	3142						
						100 000→	30 687	6551						
	24	10	Parallel	47 500	(0.375)	(0.400)	(0.500)	(0.550)	(0.595)	(0.650)				
					100 000→	100 000→	5921	1567	1725	693				
					100 000→	100 000→	7180	1722	1740	1047				
					100 000→	100 000→	7597	2179	1756	1273				
						100 000→		5854		1987				
						100 000→								
High brass Commercial........	24	0	Parallel	41 100	(0.300)	(0.350)	(0.400)	(0.500)	(0.600)	(0.700)				
					100 000→	6220	2033	746	410	343				
					100 000→	8071	2143	803	468	343				
					100 000→	13 365	2172	818	471	343				
					100 000→	28 614	2261	879	495	399				
					100 000→	50 095	3312	1001	497	444				
	24	4	Parallel	45 000	(0.300)	(0.400)	(0.500)	(0.600)	(0.700)					
					100 000→	3404	1299	748	208					
					100 000→	3687	1647	756	349					
					100 000→	10 061	1812	823	493					
					100 000→	33 090	1980	1000	505					
					100 000→	100 000→	2022	1150	620					
	24	8	Parallel	44 300	(0.400)	(0.450)	(0.500)	(0.600)	(0.700)					
					82 822	3063	1906	945	704					
					100 000→	3418	2090	1068	705					
					100 000→	5868	2279	1152	705					
					100 000→	8253	2741	1277	835					
					100 000→	26 342	2986	1439	1845					
High brass Hot rolled rolling series............	24	0	Parallel	51 400	(0.250)	(0.350)	(0.400)	(0.600)	(0.700)					
					100 000→	1522	1008	296	120					
					100 000→	2470	1135	296	122					
					100 000→	2639	1294	400	138					
						2639								

	24	4	Parallel	51 770	(0.250) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.300) 2137 15 099 17 885 51 344 100 000→	(0.335) 3110 3294 3554 5161 24 343	(0.350) 2375 3332 3332 31 301	(0.390) 1981 2054 2595 4152 5856	(0.500) 463 668 678	(0.510) 462 524 599 599 743	(0.550) 267 416 598 619	(0.620) 396 496 496 504 796	(0.650) 403 410 433 492 883	
	24	10	Parallel	50 000	(0.390) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.400) 8160 22 095 35 614 100 000→	(0.500) 1399 1765 1868 1897 2072	(0.550) 673 1340 1626	(0.600) 601 601 996 1416 1922	(0.650) 693 1047 1273 1335 1987	(0.700) 599 632 632 764 771	(0.750) 513 553 553 596 596	(0.800) 348 356 383 395 500	(0.850) 234 326 406 440 488	
Alloy G brass Rolling series........	24	0	Parallel	46 200	(0.300) 6241 14 601 15 437 23 705 32 563	(0.335) 2974 2974 4145	(0.390) 1734 1867 2274 2471 2406	(0.460) 654 654 741	(0.515) 497 762 2090	(0.540) 351 351 503	(0.610) 446 469 559				
	24	4	Parallel	46 300	(0.335) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.350) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.400) 92 871→ 100 000→ 100 000→ 100 000→ 100 000→	(0.440) 4325 4516 6692 8010 33 669	(0.490) 4174 6114 7544 13 529 16 338	(0.515) 829 1987 2088	(0.540) 1282 1600 1693	(0.610) 786 964 1098	(0.650) 604 656 699 719 797	(0.750) 352 392 392 465 465	(0.850) 189 276 286 293 293
	24	10	Parallel	43 500	(0.435) 100 000→ 100 000→ 100 000→ 100 000→	(0.460) 5459 88 738 100 000→ 100 000→ 100 000→	(0.515) 2029 4720 5695 13 985	(0.540) 1502 1622 1673 1894	(0.610) 1366 1530 1979 2459	(0.650) 1411 1411 1480 1480 1608	(0.700) 948 1107 1164 1200 1244	(0.750) 315 682 682 722 961	(0.850) 427 454 518 610 643		
Alloy G brass Commercial........	24	0	Parallel	48 300	(0.300) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.350) 6547 9164 9368 16 358 69 893→	(0.400) 1236 1247 1439 1482 1824	(0.500) 452 506 614 626 696	(0.600) 344 414 419 426 620	(0.700) 188 199 221 227 253					
	24	4	Parallel	48 400	(0.400) 5131 53 980 100 000→ 100 000→ 100 000→	(0.500) 1698 1702 1749 1956 2446	(0.600) 705 715 723 872 872	(0.700) 413 488 493 515 766							
	24	8	Parallel	45 500	(0.450) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.500) 2110 3873 4197 31 476 33 386	(0.600) 1227 1365 1492 1682 2021	(0.700) 690 737 748 785 973							

TABLE III—*Continued.*

Material	B.& S. Gage	B.& S. Numbers Hard	Direction of Rolling with Respect to Length of Specimen	Unit Stress for 1-in. Deflection, lb. per sq. in.	Number of Cycles of Reversed Stress in Thousands at Deflection in Inches Indicated in Parentheses										
Alloy B nickel silver Rolling series.......	24	0	Parallel	59 000	(0.300) 16 707 49 958 63 874 98 850 100 000→	(0.335) 13 839 18 900	(0.370) 1954 5762 6737	(0.450) 1725 1725	(0.490) 544 659 659 1320	(0.504) 805 906 987 1010	(0.569) 772 1002 1050	(0.650) 169 296 296 314			
	24	4	Parallel	60 500	(0.330) 100 000→ 100 000→ 100 000→ 100 000→	(0.364) 2816 2882 5193 5713 35 988	(0.400) 1301 1627 2834	(0.450) 1725 1725 2811	(0.504) 786 806 1103	(0.565) 578 817 936	(0.650) 303 364 434 502 649	(0.700) 288 321 339 391	(0.750) 259 259 259 365	(0.800) 134 137 144 174	(0.850) 98 143 171 332
	24	10	Parallel	58 700	(0.335) 100 000→ 100 000→ 100 000→	(0.364) 14 899 19 360 54 097 91 804 100 000→	(0.375) 7426 9189 19 673 26 133 64 262	(0.400) 3596 5163 6048 9485	(0.450) 2757 2811 2855 8142	(0.504) 987 1103 1127 1788	(0.565) 987 1003 1283 1406	(0.600) 996 996 996 996	(0.650) 427 495 667	(0.700) 358 366 374 448	(0.750) 248 248 369

Material	B.& S. Gage	B.& S. Numbers Hard	Heat Treatment	Grain Size, Ready-to-Finish Anneal, mm.	Direction of Rolling with Respect to Length of Specimen	Unit Stress for 1-in. Deflection, lb. per sq. in.	Number of Cycles of Reversed Stress in Thousands at Deflection in Inches Indicated in Parentheses					
Alloy B nickel silver Commercial.........	24	0	....	....	Parallel	54 200	(0.250) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.300) 7100 7262 7979 8173 11 620	(0.400) 2026 2026 5026 5026	(0.500) 668 952 1075 1317 1562	(0.600) 264 325 328 340 368	(0.700) 136 148 187 230 252
	24	6	....	....	Parallel	61 700	(0.300) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.350) 4766 5713 7055 13 675 42 275	(0.400) 3424 3750 5257 11 643	(0.500) 1355 1557 1582 1636 1736	(0.600) 493 577 696 720 745	(0.700) 307 308 319 358
	24	8	....	....	Parallel	64 100	(0.350) 75 391 86 074 90 873 92 366 100 000→	(0.400) 3453 10 582 13 249 43 291 49 000	(0.500) 812 1062 1172 1190 1247	(0.600) 567 651 745 752 757	(0.700) 204 339 420 431 442	

Alloy B nickel silver Commercial........	24	0	....	....	Parallel	56 200	(0.250) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.300) 5180 5300 13 510 14 027 17 194	(0.350) 1471 2895 3561 4328 4372	(0.400) 1173 1407 1781 1809 1934	(0.500) 475 526 641 1283 1411	(0.550) 289 306 317 320 330	(0.600) 190 213 348 447 447	(0.650) 173 248 248 290 447
	24	0	....	....	45 deg.	60 500	(0.250) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.300) 16 313 35 274 57 840 100 000→ 100 000→	(0.350) 2875 3208 3381 4109 8790	(0.400) 1094 1283 1507 1598 1768	(0.500) 580 644 746 746 779	(0.550) 149 285 295 323 323	(0.600) 194 194 237 237 237	(0.650) 170 254 254 410 410
	24	0	....	....	90 deg.	63 800	(0.250) 17 799 100 000→ 100 000→ 100 000→ 100 000→	(0.300) 5220 7571 7597 8436 16 267	(0.350) 2883 3184 3855 3906 3924	(0.400) 898 1035 1160 1248 1383	(0.500) 375 375 483 584 704	(0.550) 205 254 270 278 329	(0.600) 180 180 183 184 184	(0.650) 155 155 202 295 295
	24	4	....	....	Parallel	59 800	(0.250) 85 675→ 100 000→ 100 000→ 100 000→ 100 000→	(0.300) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.350) 4670 8221 9258	(0.400) 1801 1804 1828 2470 2625	(0.500) 842 929 1723 1785 1854	(0.550) 477 492 534 695 709	(0.600) 409 451 451 451 535	(0.650) 258 356 356 708 718
	24	4	....	....	45 deg.	64 400	(0.350) 6 214 10 605 100 000→ 100 000→ 100 000→	(0.400) 3632 3962 5178 5335 6484	(0.500) 1071 1342 1423 1490 1543	(0.550) 541 760 873 1041 1074	(0.600) 524 524 582 649 649	(0.650) 514 524 595 610 700		
	24	4	....	....	90 deg.	56 200	(0.250) 71 615→ 71 615→ 74 082→ 85 675→ 85 675→	(0.300) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.350) 5362 5404 6216 6789 21 472	(0.400) 2285 2734 2771 3540 3776	(0.500) 648 696 814 839 1034	(0.550) 519 552 572 641 759	(0.600) 409 409 409 467 659	(0.650) 375 375 386 534 832
	24	10	....	....	Parallel	52 050	(0.400) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.450) 100 000→ 100 000→ 100 000→ 100 000→	(0.500) 4277 4363 6697 7992 9034	(0.550) 1229 2085 2178 3743 9361	(0.600) 907 1460 1460 1783 1783	(0.650) 706 787 1204 1314	(0.700) 710 735 752 755 857	(0.800) 399 421 466 482 596
	24	10	....	....	45 deg.	49 600	(0.400) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.450) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.500) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.550) 3420 3485 8198 22 853 79 442→	(0.600) 2657 3179 3469 4010 4975	(0.700) 1646 2182 2634 2814	(0.750) 1063 1063 1150 1150 1356	(0.800) 759 952 971 1136
	24	10	....	....	90 deg.	56 700	(0.400) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.450) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.500) 35 955 59 975 79 983 100 000→ 100 000→	(0.550) 4326 6042 7410 10 886 15 095	(0.600) 1721 1721 1922 2499 2586	(0.700) 1322 1408 1596 1667 1685	(0.750) 744 795 902 902 1460	(0.800) 594 653 667 770 1016

TABLE III—*Continued.*

Material	B.& S. Gage	B.& S. Numbers Hard	Heat Treatment	Grain Size, Ready-to-Finish Anneal, mm.	Direction of Rolling with Respect to Length of Specimen	Unit Stress for 1-in. Deflection, lb. per sq. in.	Number of Cycles of Reversed Stress in Thousands at Deflection in Inches Indicated in Parentheses					
Alloy B nickel silver Special..............	24	6	....	0.017	Parallel	58 000	(0.350) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.400) 100 000→ 100 000→ 100 000– 100 000→	(0.450) 25 422 100 000→ [illegible] 000→ 100 000→ 100 000→	(0.500) 3077 19 017 31 908	(0.600) 630 842 847 1033 1255	(0.700) 342 446 459 561
	24	6	330 C., ½ hr.	0.017	Parallel	60 200	(0.350) 100 000→ 100 000→ 100 000→ 100 000→	(0.400) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.450) 1862 2741 4133 19 689 100 000→	(0.500) 1279 1593 1788 2960 3716	(0.600) 448 600 600 653 991	(0.700) 235 386 387 410 450
	24	6	....	0.022	Parallel	58 800	(0.350) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.400) 48 167 87 042 100 000→ 100 000→ 100 000→	(0.450) 3827 17 290 18 000	(0.500) 1377 1565 1633 1650 1786	(0.600) 637 673 690 695 951	
	24	6	330 C., ½ hr.	0.022	Parallel	61 000		(0.400) 46 321 89 264 100 000→ 100 000→ 100 000→	(0.450) 2321 2392 2503 3226	(0.500) 1355 1449 1708 2139	(0.600) 613 725 818 1142	(0.700) 312 317 373 438
	24	6	....	0.090	Parallel	58 100	(0.350) 100 000→ 100 000→ 100 000→ 100 000→	(0.400) 10 323 100 000→ 100 000→ 100 000→ 100 000→		(0.500) 801 1220 1640 2010 2203	(0.600) 648 657 702 943	(0.700) 285 342 391 571
	24	6	330 C., ½ hr.	0.090	Parallel	56 900		(0.400) 9569 100 000→ 100 000→	(0.450) 2263 2606 3398	(0.500) 925 1087 1763 2253	(0.600) 584 681 682 714	(0.700) 300 454 454 454

Alloy B nickel silver Special..............	24	8	....	0.015	Parallel	58 100	(0.400) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.450) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.500) 5969 14 719 16 398 16 858 18 201	(0.600) 819 951 1038 1103 1228	
	24	8	330 C., ½ hr.	0.015	Parallel	59 500	(0.400) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.450) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.500) 2471 4284 5428 5845	(0.600) 1052 1419 1561 1761	(0.700) 352 355 462 475
	24	8	....	0.020	Parallel	54 300	(0.400) 100 000→ 100 000→ 100 000→ 100 000→	(0.450) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.500) 10 682 11 368 26 388 51 324	(0.600) 828 1327 1861	(0.700) 444 515 545 655
	24	8	330 C., ½ hr.	0.020	Parallel	60 200	(0.400) 100 000→ 100 000→ 100 000→ 100 000→	(0.450) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.500) 3255 4513 29 476	(0.600) 734 1167 1305 1617	(0.700) 444 444 545 545
	24	8	....	0.090	Parallel	60 400	(0.350) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.400) 4002 4662 8843 11 997 65 652	(0.500) 991 1046 1264 1368 1585	(0.600) 525 547 644 827	(0.700) 289 308 316 350
	24	8	330 C., ½ hr.	0.090	Parallel	62 430	(0.350) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.400) 5280 8026 11 817 12 284 19 387	(0.500) 699 824 910 1199	(0.600) 391 476 510 543	(0.700) 236 288 290 545
Alloy B nickel silver $\frac{1}{32}$ by $\frac{1}{32}$ in. square hole	24	6			Parallel	67 500	(0.250) 100 000→ 100 000→ 100 000→	(0.300) 21 715 42 003 82 990 100 000→	(0.350) 1940 2506 3671 3956	(0.400) 1283 2274 2511 2719	(0.500) 591 663 879 976
$\frac{1}{32}$ by $\frac{3}{64}$ in. rectangular hole..............	24	6			Parallel	79 430	(0.200) 7258 100 000→ 100 000→ 100 000→	(0.250) 3392 4617 6926 10 769	(0.300) 2412 3132 3278 4893	(0.400) 713 983 999 1406	(0.500) 301 337 374 404
$\frac{1}{32}$ by $\frac{1}{16}$ in. rectangular hole..............	24	6			Parallel	89 570	(0.200) 26 145 100 000→ 100 000→ 100 000→	(0.250) 1453 2244 3054 6323	(0.300) 968 1674 1941 2422	(0.400) 413 422 521 653	

TABLE III—*Continued.*

Material	B. & S. Gage	B. & S. Numbers Hard	Heat Treatment	Grain Size, Ready-to-Finish Anneal, mm.	Direction of Rolling with Respect to Length of Specimen	Unit Stress for 1-in. Deflection, lb. per sq. in.	Number of Cycles of Reversed Stress in Thousands at Deflection in Inches Indicated in Parentheses						
Alloy B nickel silver (continued) 0.001 in. scratch on one side..............	24	6			Parallel	54 000	(0.300)	(0.350)	(0.400)	(0.450)	(0.500)	(0.600)	(0.700)
							100 000→	100 000→	100 000→	2258	3362	1157	221
							100 000→	100 000→	100 000→	11 337	8041	1197	235
							100 000→	100 000→	100 000→	17 982	36 506	1692	338
							100 000→	100 000→	100 000→	21 180	37 294	1929	394
90 deg. bend—$3\frac{1}{32}$-in. radii..............	24	6			Parallel	50 000	(0.200)	(0.250)→	(0.300)	(0.350)	(0.400)	(0.500)	
							100 000→	100 000→	100 000→	1073	368	300	
							100 000→	100 000→	100 000→	1556	462	469	
							100 000→	100 000→	100 000→	1700	565	849	
							100 000→	100 000→	100 000→	1845	1102	852	
							100 000→		100 000→	40 086	4557		
$\frac{1}{8}$-in. offset—$\frac{1}{32}$-in. radii.	24	6			Parallel	51,020	(0.250)	(0.400)	(0.600)				
							100 000→	2511	465				
							100 000→	3866	472				
							100 000→	3974	582				
							100 000→	5409	627				
Copper—Commercial...	24	0	....	....	Parallel	45 000	(0.200)	(0.250)	(0.300)	(0.400)	(0.500)		
							100 000→	4214	4373	369	115		
							100 000→	4558	5323	353	157		
							100 000→	4848	5323	891	181		
							100 000→	5191	5388		201		
							100 000→	5539	7804		221		
	24	2	....	....	Parallel	52 200	(0.200)	(0.250)	(0.275)	(0.300)	(0.400)	(0.500)	(0.600)
							100 000→	100 000→	21 262	8935	1354	324	131
							100 000→	100 000→	41 009	10 785	1617	337	132
							100 000→	100 000→		12 758	2188	358	141
							100 000→	100 000→		26 057	2276	406	152
								100 000→		61 629	2984	617	169
	24	6	....	....	Parallel	44 700	(0.300)	(0.350)	(0.400)	(0.500)	(0.600)		
							20 171	3678	1460	156	133		
							27 766	5547	2439	223	143		
							100 000→	6108	3819	291	144		
							100 000→	6928	4603	352	155		
							100 000→	40 124	4751	405	172		
Everdur—rolling series..	24	8	....		Parallel	50 000	(0.465)	(0.480)	(0.500)	(0.515)	(0.620)		
							100 000→	4449	2651	3058	1050		
							100 000→	8663	3039	3535	1187		
							100 000→	17 000→	4906	3821			
							100 000→	17 100→	5792	4482			
							100 000→	17 100→	11 141	6295			

Everdur—Commercial, first lot	24	8	....		Parallel	50 800	(0.450) 100 000→ 100 000→ 100 000→ 100 000→	(0.500) 2943 23 265 25 785 100 000→ 100 000→	(0.600) 1669 2338 5788			
Everdur—Commercial, second lot	24	0	....		Parallel	46 200	(0.300) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.350) 12 642 18 400 19 734 20 285 21 228	(0.400) 2738 2784 3224 3356 5536	(0.500) 524 675 750 859 916	(0.600) 357 377 378 401 405	(0.700) 192 193 195 202 210
	24	4	....		Parallel	44 900	(0.400) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.500) 5757 5938 8827 76 593 100 000→	(0.600) 3118 3689 3694 3874	(0.700) 1365 1390 1780 1889 1954		
	24	8	....		Parallel	42 500	(0.400) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.500) 5522 5373 34 860 100 000→ 100 000→	(0.600) 1207 3066 5497 5715 6259	(0.700) 955 955 1100 1100 1100		

Material	B. & S. Gage	B. & S. Numbers Hard	Finish	Direction of Rolling with Respect to Length of Specimen	Unit Stress for 1-in. Deflection, lb. per sq. in.	Number of Cycles of Reversed Stress in Thousands at Deflection in Inches Indicated in Parentheses					
Alloy A phosphor bronze Commercial	24	0	....	Parallel	46 900	(0.300) 6490 33 378 100 000→ 100 000→ 100 000→	(0.400) 4028 4590 5235 5424 9464	(0.500) 207 255 268 538	(0.600) 151 169 188 192 209	(0.700) 50 57 186 186 195	
	24	4	....	Parallel	51 700	(0.300) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.350) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.500) 14 631 17 200 100 000→ 100 000→ 100 000→	(0.600) 423 2549 2858 18 181 18 959	(0.700) 695 718 839 980 1052	
	24	8	....	Parallel	45 300	(0.400) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.450) 100 000→ 100 000→	(0.500) 4615 13 580 24 415 100 000→ 100 000→	(0.550) 13 061	(0.600) 1378 1434 2078 2477 6486	(0.700) 504 613 710 732 788

TABLE III—*Continued.*

Material	B. & S. Gage	B. & S. Numbers Hard	Finish	Direction of Rolling with Respect to Length of Specimen	Unit Stress for 1-in. Deflection, lb. per sq. in.	Number of Cycles of Reversed Stress in Thousands at Deflection in Inches Indicated in Parentheses					
Alloy C phosphor bronze Rolling series	24	0		Parallel	43 100	(0.390) 100 000→ 100 000→	(0.490) 2347 2463 2870 9023	(0.510) 1638 2725 5121 7629	(0.610) 885 947 988	(0.830) 111 162 277	
	24	4		Parallel	41 200	(0.390) 100 000→ 100 000→	(0.535) 80 556 100 000→ 100 000→ 100 000→	(0.560) 1931 9234 11 278 13 927 37 516	(0.610) 2380 2638 2787	(0.620) 2221 3007 3007 8126	(0.830) 578 709 772 840
	24	10		Parallel	43 500	(0.390) 100 000→	(0.560) 80 610 100 000→ 100 000→ 100 000→ 100 000→	(0.600) 10 864 13 515 16 577 23 975 32 490	(0.700) 1813 1877 2031 3452	(0.830) 601 958 1090	
Alloy C phosphor bronze Commercial	24	0		Parallel	42 900	(0.300) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.400) 12 660 14 724 39 830 47 960 55 983	(0.500) 1687 3903 3903 4279 5279	(0.600) 431 499 501 566 953	(0.700) 112 161 171 205 229	
	24	4		Parallel	45 500	(0.450) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.500) 6948 15 687 59 733 78 853 79 455	(0.600) 3121 4009 4172 5180 5864	(0.700) 926 1083 1115 1150 1154		
	24	8		Parallel	44 200	(0.350) 100 000→ 100 000→ 100 000→	(0.450) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.500) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.550) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.600) 28 268 30 854 46 765 57 854→ 57 854→	(0.700) 1600 1601 1618 3084 4518
Alloy C phosphor bronze Commercial	24	8	Unfinished Control Specimens	Parallel	44 600	(0.438) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.488) 100 000→ 100 000→ 100 000→ 100 000→	(0.538) 100 000→ 100 000→ 100 000→	(0.588) 25 121 26 018 74 543 100 000→ 100 000→	(0.638) 1509 1851 2245 13 759 17 700	(0.663) 1316 1840 2072 2092 2838

24	8	Finish A	Parallel	44 600	(0.388) 100 000→ 100 000→ 100 000→ 100 000→	(0.438) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.488) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.538) 19 683 100 000→ 100 000→ 100 000→ 100 000→	(0.588) 2969 3570 3612 3890	(0.638) 1640 1648 1975 1998
24	8	Finish B	Parallel	44 600	(0.388) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.438) 100 000→ 100 000→ 100 000→ 100 000→	(0.488) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.538) 2161 5305 21 784 100 000→ 100 000→	(0.588) 3614 11 672 44 148 60 841	(0.638) 2088 2490 2564 2727
24	8	Finish C	Parallel	44 600	(0.388) 100 000→ 100 000→ 100 000→ 100 000→	(0.438) 5826 12 607 38 829 69 289	(0.488) 2919 4992 6108 9224	(0.538) 2440 5457 5956 6457 6792	(0.588) 1263 1860 1860 3170 3367	(0.638) 1336 1383 1553 1853 1864
24	8	Finish D	Parallel	44 600	(0.588) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.638) 1062 1744 16 716 26 533 32 300	(0.700) 1088 1747 17 700 23 797			
24	8	Finish E	Parallel	44 600	(0.388) 87 600→ 87 600→ 87 600→ 87 600→ 87 600→	(0.438) 87 600→ 100 000→ 100 000→	(0.488) 100 000→ 100 000→ 100 000→ 100 000→	(0.538) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.588) 24 389 25 344 50 632 51 038	(0.638) 1323 19 728 23 334 24 305 28 141
24	8	Finish F	Parallel	44 600	(0.388) 100 000→ 100 000→ 100 000→	(0.500) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.588) 6739 21 080 25 739 39 293	(0.638) 1827 2060 3010 3836 5531	(0.700) 1000 1026 1224 1341 1443	
24	8	Microscopic Polish	Parallel	44 600	(0.450) 100 000→ 100 000→	(0.500) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.600) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.650) 18 037 43 250 48 349 100 000→ 100 000→	(0.700) 8767	(0.800) 1552 1598 1916 3334

TABLE III.—*Continued.*

Material	B.&S. Gage	B.&S. Numbers Hard	Heat Treatment	Direction of Rolling with Respect to Length of Specimen	Unit Stress for 1-in. Deflection, lb. per sq. in.	Number of Cycles of Reversed Stress in Thousands at Deflection in Inches Indicated in Parentheses				
Beryllium-copper Lot P.......	18	0	Annealed at 800 C. Quenched in water. Aged 2 hr. at 275 C.	Parallel	103 900	(0.300) 18 718 52 537 68 600	(0.350) 1 391 1 948 5 546	(0.425) 499 1 009 1 208 2 424	(0.536) 365 365 624 1 234	
Lot OP.....	18	0	Annealed at 800 C. Quenched in water. Aged 2 hr. at 275 C.	Parallel	94 300	(0.300) 9 890 88 064 100 000→	(0.350) 4 942 5 898 17 082	(0.425) 365 365 624		
Lot 2P......	18	2	Annealed at 800 C. Quenched in water. Cold rolled. Aged 2 hr. at 275 C.	Parallel	90 900	(0.275) 100 000→ 100 000→ 100 000→	(0.300) 51 289 100 000→ 100 000→	(0.350) 2 696 3 384 4 031	(0.425) 1 393 1 598 1 954 3 611	(0.536) 624 722 1 053 1 126
Lot 4P......	18	4	Annealed at 800 C. Quenched in water. Cold rolled. Aged 2 hr. at 275 C.	Parallel	92 200	(0.300) 92 238 100 000→ 100 000→	(0.350) 12 468 44 446 58 464→	(0.425) 1 613 3 062 4 368 20 815	(0.536) 741 741 868 1 226	(0.634) 204 410 641 752
eryllium-copper Lot 5.......	24	4	Annealed at 800 C. Quenched in water. Cold rolled.	Parallel	45 000	(0.500) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.600) 50 727 100 000→ 100 000→ 100 000→ 100 000→	(0.700) 9 899 13 191 36 436 43 208 58 355	(0.750) 1 202 1 234 1 667 2 619 8 277	(0.800) 573 654 1 168 1 319 2 130
Lot 5—aged..	24	4	Annealed at 800 C. Quenched in water. Cold rolled. Aged 2 hr. at 275 C.	Parallel	51 800	(0.500) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.600) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.700) 25 570 25 815 27 034 31 150 36 159	(0.750) 3 778 7 130 11 230 18 061 20 980	(0.800) 2 712 3 810 3 838 4 204 4 270
Lot 6.......	18	2	Annealed at 800 C. Quenched in water. Cold rolled.	Parallel	93 800	(0.200) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.250) 10 160 24 546 25 550 26 315 100 000→	(0.300) 801 820 923 1 157 1 976	(0.500) 90 94 107 108 112	

Lot 6—aged..	18	2	Annealed at 800 C. Quenched in water. Cold rolled. Aged 2 hr. at 275 C.	Parallel	102 900	(0.200) 100 000→ 100 000→ 100 000→ 100 000→	(0.250) 34 090 53 145 64 678 100 000→ 100 000→	(0.300) 10 489 15 848 20 728 30 972 41 724	(0.400) 917 1 058 1 230 2 099 2 217	(0.500) 224 226 250 269 320				
Lot 7.......	24	2	Annealed at 800 C. Quenched in water. Cold rolled.	Parallel	46 900	(0.500) 45 333 77 766 100 000→ 100 000→	(0.600) 1 125 1 223 1 767 1 927 2 087	(0.700) 617 627 659 690 725						
Lot 7—aged..	24	2	Annealed at 800 C. Quenched in water. Cold rolled. Aged 2 hr. at 275 C.	Parallel	53 500	(0.500) 32 890 61 791 72 118 94 342 94 801	(0.600) 3 507 3 682 5 576 10 001 55 705	(0.700) 1 997 2 341 2 378 2 817 2 978						
Lot 7—aged..	24	2	Annealed at 800 C. Quenched in water. Cold rolled. Aged 1 hr. at 275 C.	Parallel	51 000	(0.450) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.500) 100 000→ 100 000 → 100 000→ 100 000→ 100 000→	(0.550) 100 000→ 100 000→ 100 000→ 100 000→	(0.600) 6 600 13 734 20 506 57 082 100 000→	(0.650) 2 399 2 726 3 429 3 438 4 178	(0.700) 2 624 2 703 3 424 3 624 3 734			
Dispersion hardened copper Alloy No. 80.	24	0	....	Parallel	39 600	(0.350) 997 75 888→ 100 000→ 100 000→	(0.490) 872 9 994 40 682 100 000→ 100 000→	(0.500) 408 520 3 330 100 000→	(0.535) 335 681 681 5 592 44 631	(0.550) 217 231 691 1797	(0.600) 169 224 524 995	(0.650) 169 183 524 644		
	24	10	....	Parallel	47 800	(0.490) 5 087 5 868 100 000→ 100 000→ 100 000→	(0.500) 3 021 35 179 39 580 75 003 100 000→	(0.535) 16 338 49 074→ 49 074→ 49 074→ 49 074→	(0.550) 2 772 2 946 3 139 49 870	(0.600) 491 665 882 1 176	(0.650) 296 491 547 1 325			
Dispersion hardened brasses Alloy No. 33.	24	....	Quenched 800 C. Aged 1 hr. at 500 C.	Parallel	49 300	(0.305) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.330) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.390) 21 259 45 854 63 552 69 792 93 357	(0.400) 2 208 3 165 3 491 20 306	(0.460) 1 400 1 970 2 249 2 352 2 707	(0.465) 717 786 877 935 1 000	(0.600) 300 324 461 700	(0.675) 234 226 350 362 608	(0.700) 134 162 191 261

TABLE III.—*Continued.*

Material	B.&S. Gage	B.&S. Numbers Hard	Heat Treatment	Direction of Rolling with Respect to Length of Specimen	Unit Stress for 1 in. Deflection, lb. per sq. in.	Number of Cycles of Reversed Stress in Thousands at Deflection in Inches Indicated in Parentheses											
Dispersion hardened brasses (continued)																	
Alloy No. 34.	24	....	Quenched 850 C. Aged 1 hr. at 500 C.	Parallel	49 500	(0.305)	(0.330)	(0.390)	(0.400)	(0.460)	(0.465)	(0.600)	(0.675)	(0.700)			
						100 000→	2 708	2 916	1 125	873	673	290	265	141			
						100 000→	3 838	3 448	2 402	1 144	984	346	286	177			
						100 000→	59 432	3 448	3 018	1 256	1 070	696	353	203			
						100 000→	79 198	3 760	3 210	1 400	1 075	696	418	210			
						100 000→	86 417	6 840	3 311	1 767	1 250	696	418	229			
Alloy No. 35.	24	....	Quenched 800 C. Aged 1 hr. at 500 C.	Parallel	45 300	(0.305)	(0.390)	(0.460)	(0.480)	(0.500)	(0.600)	(0.675)	(0.700)				
						100 000→	100 000→	2 100	965	952	659	740	359				
						100 000→	100 000→	2 576	1 425	1 227	815	740	377				
						100 000→	100 000→	63 247	2 062	1 860	867	883	463				
						100 000→	100 000→	69 386	2 475	1 869	887	971	498				
						100 000→	100 000→	100 000→	2 475	2 292	1 001	1 044	512				
Alloy No. 79.	24	10	Heated 1 hr. at 800 C. Quenched in water. Aged 2 hr. at 400 C.	Parallel	43 720	(0.290)	(0.335)	(0.375)	(0.440)	(0.510)	(0.560)	(0.600)	(0.650)	(0.700)	(0.750)	(0.800)	(0.850)
						100 000→	100 000→	100 000→	4 077	699	1 808	661	86 679	1 273	852	599	440
						100 000→	100 000→	100 000→	10 209	54 594	2 461	11 646	92 649	1 341	1 242	726	450
						100 000→	100 000→	100 000→	17 616	100 000→	100 000→	32 104	100 000→	3 097→	1 690	916	598
						100 000→	100 000→	100 000→	100 000→	100 000→	100 000→	70 891	100 000→	3 097→	3 563	1 619	
Alloy No. 85.	24	0	Heated 1 hr. at 800 C. Quenched in water. Aged 2 hr. at 400 C.	Parallel	47 800	(0.250)	(0.300)	(0.350)	(0.400)	(0.550)	(0.600)	(0.650)	(0.700)	(0.750)			
						100 000→	99 263→	31 570	3 394	411	296	323	256	66			
						100 000→	99 263→	51 969	3 771	421	491	452	260	66			
						100 000→	100 000→	100 000→	5 613	665	547	571	325	231			
						100 000→	100 000→	100 000→	47 912	786	728	1 267	491	232			
						100 000→	100 000→	100 000→	83 775	786	902	1 267	643	396			
Alloy No. 77.	24	4	Heated 1 hr. at 800 C. Quenched in water. Aged 2 hr. at 400 C.	Parallel	46 400	(0.290)	(0.335)	(0.375)	(0.400)	(0.440)	(0.510)	(0.560)	(0.600)	(0.650)	(0.750)	(0.850)	
						100 000→	100 000→	100 000→	100 000→	8 649	2 725	2 462	528	1 384	852	498	
						100 000→	100 000→	100 000→	100 000→	28 280	5 250	10 220	1 806	2 193	1 435	735	
						100 000→	100 000→	100 000→	100 000→	100 000→	100 000→	20 207	3 433	4 172	1 487	763	
						100 000→	100 000→	100 000→	100 000→	100 000→	100 000→	74 987	11 152	100 000→	1 690	1 109	
	24	10	Heated 1 hr. at 800 C. Quenched in water. Aged 2 hr. at 400 C.	Parallel	43 480	(0.290)	(0.335)	(0.375)	(0.440)	(0.510)	(0.550)	(0.560)	(0.600)	(0.650)	(0.750)	(0.850)	
						100 000→	100 000→	100 000→	100 000→	4 045	10 365	8 666	8 127	91 097	4 558	750	
						100 000→	100 000→	100 000→	100 000→	100 000→	74 179	14 022	8 867	100 000→	5 064	1 262	
						100 000→	100 000→	100 000→	100 000→	100 000→	96 891	29 428	70 891	100 000→	5 377	2 530→	
						100 000→	100 000→	100 000→	100 000→	100 000→		35 849					
Alloy No. 78.	24	0	Heated 1 hr. at 800 C. Quenched in water. Aged 2 hr. at 400 C.	Parallel	46 900	(0.335)	(0.375)	(0.435)	(0.510)	(0.560)	(0.600)	(0.650)	(0.750)	0.850)			
						100 000→	100 000→	91 791→	2 667	1 928	1 353	713	588	357			
						100 000→	100 000→	93 644→	5 626	4 766	1 428	1 607	621	418			
						100 000→	100 000→	94 771→	28 736	4 844	2 131	1 986	879	465			
						100 000→	100 000→	94 771→	30 580	6 214	3 538	2 435	1 074	490			
	24	4	Heated 1 hr. at 800 C. Quenched in water. Aged 2 hr. at 400 C.	Parallel	47 600	(0.290)	(0.335)	(0.400)	(0.440)	(0.510)	(0.560)	(0.650)	(0.750)	(0.850)			
						100 000→	100 000→	100 000→	20 507	10 448	2 668	3 265	1 690	424			
						100 000→	100 000→	100 000→	64 738	100 000→	62 270	100 000→	2 924	600			
						100 000→	100 000→	100 000→	100 000→	100 000→	100 000→	100 000→	3 792	626			
						100 000→	100 000→		100 000→	100 000→	100 000→	100 000→	20 118	669			
	24	10	Heated 1 hr. at 800 C. Quenched in water. Aged 2 hr. at 400 C.	Parallel	46 580	(0.375)	(0.400)	(0.510)	(0.560)	(0.600)	(0.650)	(0.700)	(0.750)	(0.800)	(0.850)		
						100 000→	100 000→	100 000→	4 027	1 979	2 193	1 005	687	718	418		
						100 000→	100 000→	100 000→	100 000→	11 519	100 000→	2 321	49 906	1 210	429		
						100 000→	100 000→	100 000→	100 000→	11 725	100 000→	8 100	49 906→	1 688→	808		
							100 000→	100 000→	100 000→		100 000→	8 100	49 906→		808		

TABLE III—*Continued*.

Material	B. & S. Gage	B. & S. Numbers Hard	Direction of Rolling with Respect to Length of Specimen	Unit Stress for 1-in. Deflection, lb. per sq. in.	Number of Cycles of Reversed Stress in Thousands at Deflection in Inches Indicated in Parentheses								
Monel metal Rolling series	22	0	Parallel	92 400	(0.250) 10 019 42 525 42 525 63 855 66 059	(0.300) 1088 1120 1193 1300 1550	(0.335) 570 626 793 1007 1363	(0.440) 198 198 198 198 198					
	22	Half Hard	Parallel	97 070	(0.250) 13 256 13 299 26 179 32 897 35 717	(0.290) 1686 2749 3116 3240 3627	(0.335) 1203 1579 2247 2636	(0.350) 573 634 865 996 1516	(0.440) 206 223 232 317 317				
	22	Full Hard	Parallel	103 700	(0.330) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.350) 100 000→ 100 000→ 100 000→ 100 000→	(0.364) 30 745 86 886 100 000→ 100 000→	(0.400) 6039 6039 6457 6457	(0.440) 1010 1284 1426 1955 2533	(0.500) 552 619 637			
Monel metal Commercial	24	0	Parallel	73 400	(0.300) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.350) 5126 13 756 14 935 14 935 15 474	(0.400) 9103 9281 9645 10 036	(0.500) 31 172 200 201 596					
	24	Hard	Parallel	81 400	(0.400) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.500) 4481 6997 7453 8687 9882	(0.600) 624 815 1013 1013 1013	(0.700) 297 314 341 418 486					
	24	Full Hard	Parallel	78 400	(0.400) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.500) 3211 3332 4034 4829 5511	(0.600) 611 948 983 1227	(0.700) 278 285 304 315 318					
High silicon monel metal	25	....	Parallel	68 400	(0.335) 100 000→ 100 000→	(0.550) 29 976 74 295 100 000→ 100 000→ 100 000→	(0.560) 62 844 100 000→ 100 000→ 100 000→	(0.600) 43 263 57 535 72 860→ 72 860→	(0.620) 32 499 62 466 72 456	(0.650) 570 8446 44 247 100 000→	(0.700) 1343 1877 2110 3904 5120	(0.750) 596 1061 1353 1522 2220	(0.800) 345 810 1280 1688

TABLE III—*Continued.*

Material	B. & S. Gage	B. & S. Numbers Hard	Direction of Rolling with Respect to Length of Specimen	Unit Stress for 1-in. Deflection, lb. per sq. in.	Number of Cycles of Reversed Stress in Thousands at Deflection in Inches Indicated in Parentheses							
K Monel metal	24	Annealed	Parallel	79 500	(0.330) 100 000→ 100 000→	(0.335) 70 643 100 000→ 100 000→	(0.350) 9345 40 917 51 312	(0.375) 4576 7988 10 202 12 795 14 439	(0.440) 1715 1716 1741 1859 1859	(0.510) 422 569 569 934	(0.560) 323 323 411	(0.620) 267 290 443
	24	Heat Treated	Parallel	83 950	(0.335) 100 000→ 100 000→ 100 000→	(0.400) 100 000→ 100 000→ 100 000→	(0.440) 20 832 48 582 66 123 95 035 100 000→	(0.560) 2700 2780 3493	(0.600) 1215 1215 1416 1416	(0.650) 714 776 1023		
Aluminum alloy 2S............	24	H	Parallel	32 250	(0.250) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.300) 6544 7468 7625 22 019	(0.350) 1066 1312 1370 3978 4281	(0.400) 553 579 657 693 715	(0.440) 168 168 277 587 874	(0.510) 107 125 214 214 218		
	24	H	45 deg.	31 100	(0.300) 4761 8456 13 820 39 315 100 000→	(0.350) 913 1928 4451 4775 9239	(0.400) 649 717 883 1080 1247	(0.440) 172 256 295 306 343	(0.510) 120 131 131 144 915			
	24	H	90 deg.	32 000	(0.250) 100 000→ 100 000→ 100 000→ 100 000→	(0.300) 2657 7241 9449 10 768	(0.350) 1007 1546 2313 2834 6916	(0.400) 378 392 440 489 519	(0.440) 231 231 313 313 587	(0.510) 132 132 132 224 224		
Aluminum alloy 3S............	24	H	Parallel	30 700	(0.250) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.300) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.350) 24 484 29 801 35 491 100 000→ 100 000→	(0.400) 895 3460 5071 5394 6755	(0.440) 513 538 627 868 868	(0.510) 234 234 360 360 869	(0.560)	
	24	H	45 deg.	29 400	(0.250) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.300) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.350) 15 207 16 243 18 858 100 000→ 100 000→	(0.400) 1781 1812 3152 3365 4814	(0.440) 637 717 939 1113 1249	(0.510) 145 254 272 316 320	(0.560) 109 278 298 300 300	

	24	H	90 deg.	30 200	(0.250) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.300) 100 000→ 100 000→ 100 000→ 100 000→	(0.350) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.400) 229 1605 2069 4035	(0.440) 473 684 1069 1069 1069	(0.510) 237 259 299 338 380
Aluminum alloy 17S..........	24	0	Parallel	32 000	(0.300) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.350) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.400) 2773 4296 6905 15 353 17 782	(0.450) 741 816 846 1123 1649	(0.500) 312 382 446 527 568	
	24	0	45 deg.	32 750	(0.350) 71 615→ 71 615→ 100 000→ 100 000→ 100 000→	(0.400) 4421 7536 12 422 13 804 15 357	(0.450) 804 856 899 1366 2125	(0.500) 283 380 388 536 780	(0.550) 129 136 141 210 236	
	24	0	90 deg.	31 850	(0.300) 100 000→ 100 000→ 100 000→ 100 000→	(0.350) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.400) 751 3326 6905 10 731 10 956	(0.450) 680 740 2370 2572 3970	(0.500) 199 276 337 472 502	
Aluminum alloy 17S..........	24	T	Parallel	30 700	(0.500) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.550) 793 12 427 13 125 58 875 84 645→	(0.600) 4995 14 723 15 041 29 273 40 333	(0.650) 1883 2679 3572 3970 4144	(0.700) 2342	(0.800) 73 153 262 284 1034
	24	T	45 deg.	30 800	(0.500) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.550) 6898 35 066 47 929 55 375 58 684	(0.600) 13 078 39 320 48 679	(0.650) 3138 5862 7837 10 340	(0.700) 765	(0.800) 347 601 624 1160
	24	T	90 deg.	30 750	(0.500) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.550) 10 256 46 350 46 350 53 624 60 246	(0.600) 20 216 21 891 38 377 100 000→ 100 000→	(0.650) 8153 15 796 25 159 56 097→ 57 054→	(0.700) 6232→	

TABLE III—*Concluded.*

Material	B. & S. Gage	B. & S. Numbers Hard	Direction of Rolling with Respect to Length of Specimen	Unit Stress for 1-in. Deflection, lb. per sq. in.	Number of Cycles of Reversed Stress in Thousands at Deflection in Inches Indicated in Parentheses					
Aluminum alloy 17S...........	24	H	Parallel	34 400	(0.350) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.400) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.450) 2163 16 187 70 114	(0.500) 831 1113 1647 2570 3235	(0.550) 190 277 314 324 437	(0.600) 157 294 294 294 294
	24	H	45 deg.	34 500	(0.350) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.400) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.450) 1876 100 000→ 100 000→ 100 000→ 100 000→	(0.500) 1105 2068 4064 24 570	(0.550) 421 610 683 720 1027	(0.650) 81 151 181 193 388
	24	H	90 deg.	35 200	(0.350) 100 000→ 100 000→ 100 000→ 100 000→ 100 000→	(0.400) 11 295 100 000→ 100 000→ 100 000→	(0.500) 1672 1815 2700 2713 3469	(0.550) 301 317 541 671 1084	(0.650) 107 151 151 180 377	
Nickel[a].....................	24	0	Parallel	91 800	(0.250) 100 000→	(0.300) 6777 10 299 10 566 10 566 10 738	(0.400) 258 444 444 573 573	(0.500) 86 87 89 155 162		
			45 deg.	88 700	(0.300) 64 348 70 000→ 70 000→	(0.350) 1742 2130 2191	(0.400) 609	(0.500) 106 114 116 138 192		
			90 deg.	75 500	(0.300) 61 314 70 000→ 70 000→	(0.350) 1325 1546 1808	(0.400) 582 705 755 834 1231	(0.500) 284 284 284 284 414		

Nickel[a]*—Cont'd*...............	24	4	Parallel	100 700	(0.350) 100 000→ 100 000→ 100 000→ 100 000→	(0.400) 34 362 85 000→ 85 000→	(0.450) 1026 1118 2316 3076 3277	(0.500) 814 828 1035 1061 1249	
		4	45 deg.	89 200	(0.400) 85 000→ 85 000→ 100 000→	(0.450) 12 604 90 000→ 90 000→	(0.500) 3627 8830 49 941 66 407	(0.600) 598 627 1019 1077 1091	
			90 deg.	98 000	(0.400) 85 000→ 85 000→ 100 000→	(0.450) 9594 14 700 16 611 16 711 20 802	(0.500) 1650 1914 2375 4386	(0.600) 293 395 402 596	(0.700) 145 178 183
	24	8	Parallel	102 820	(0.400) 33 864 85 000→ 85 000→	(0.450) 1809	(0.500) 1783 2073		
			45 deg.	85 400	(0.400) 85 000→ 85 000→ 100 000→	(0.450) 25 874 35 000→ 35 000→	(0.500) 20 394 28 395	(0.600) 1593 3595	(0.700) 364 365 385 403
			90 deg.	102 820	(0.400) 85 000→ 85 000→ 100 000→	(0.450) 4805 26 704	(0.500) 1783 2073 75 000→ 75 000→		

NOTE: → Specimen Unbroken.

[a] These tests are not as yet completed.

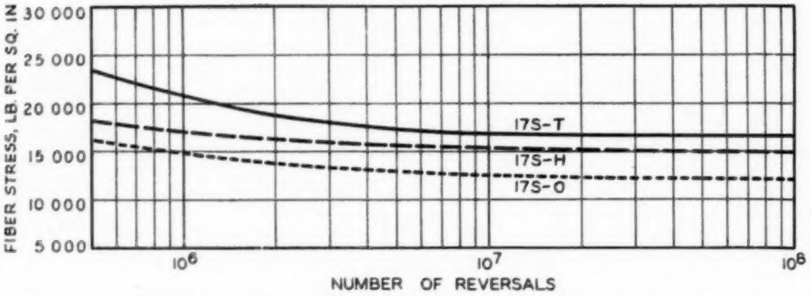

FIG. 4.—Relation of Fiber Stress to Reversal of Stress, Aluminum Alloy No. 17S.

No. 24 B. & S. Gage. Average curves. Grain direction parallel to length.

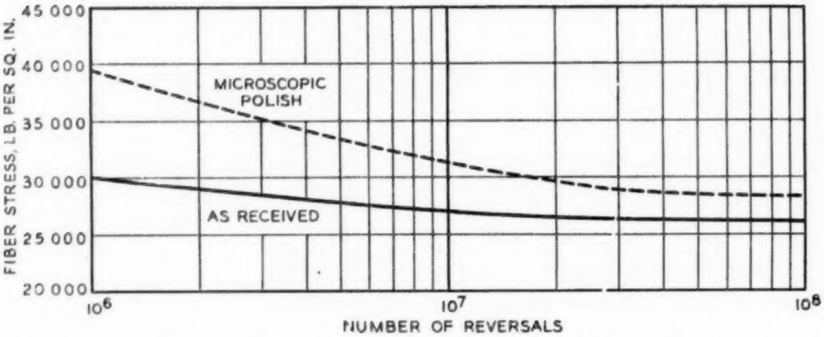

FIG. 5.—Effect of Surface Preparation on Fatigue Properties, Alloy C Phosphor Bronze.

No. 24 B. & S. Gage, 8 numbers hard. Average curves. Grain direction parallel to length.

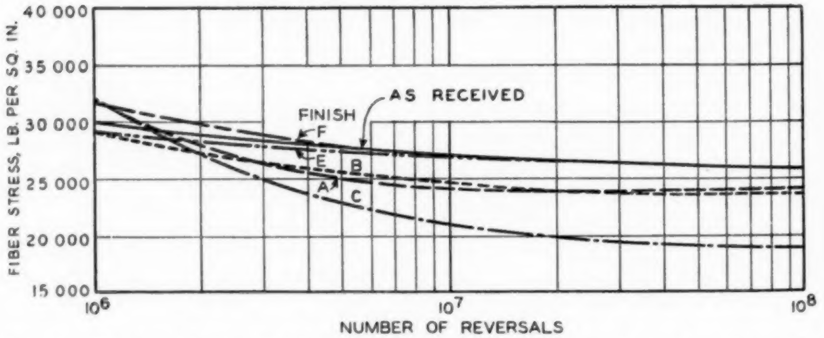

FIG. 6.—Effect of Various Finishes and Surface Preparations on the Fatigue Properties of Alloy C Phosphor Bronze.

No. 24 B. & S. Gage, 8 number hard. Average curves. Grain direction parallel to length.

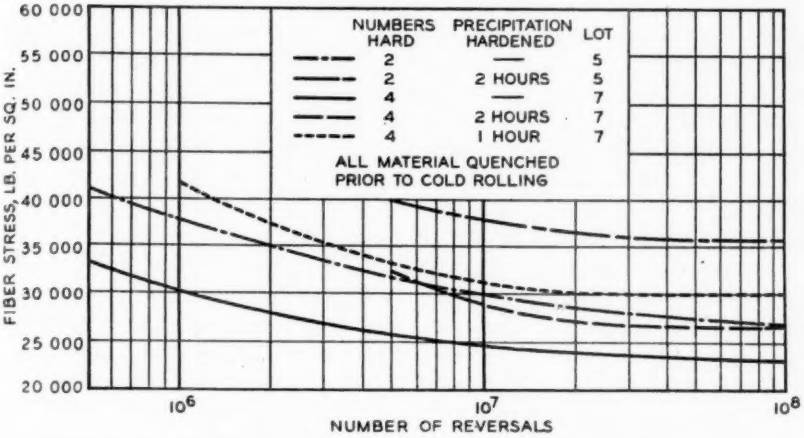

FIG. 7.—Relation of Fiber Stress to Reversal of Stress. 0.5 per cent Nickel, 2.25 per cent beryllium, beryllium-copper alloy.

No. 24 B. & S. Gage. Average curves. Grain direction parallel to length.

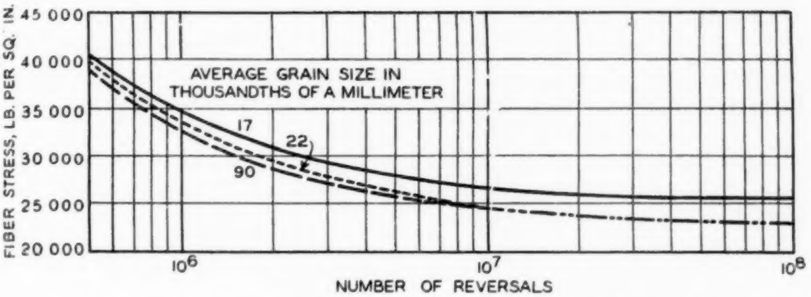

FIG. 8.—Effect of Grain size on Fatigue Properties of Alloy B Nickel Silver.

No. 24 B. & S. Gage, 6 numbers hard. Average curves. Grain direction parallel to length.

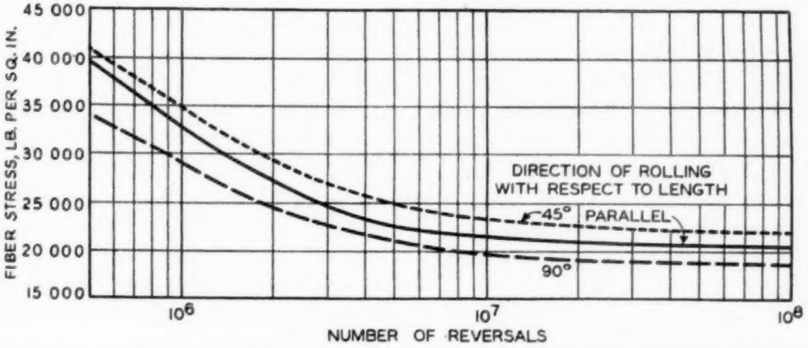

FIG. 9.—Effect of Grain Direction on Fatigue Properties of Alloy B Nickel Silver.

No. 24 B. & S. Gage, 4 numbers hard. Average curves.

the endurance limit reported for a given number of cycles of reversed stress will not necessarily hold for a larger number of cycles. In our work, however, we have considered 100,000,000 cycles of reversed stress to be the endurance limit.

The data plotted in Fig. 4 show that the endurance properties of aluminum alloy 17S can be definitely improved by hard working and that even further improvement will result if this alloy is heat treated.

The data plotted in Fig. 5 show that considerable improvement in the fatigue characteristics may be obtained by careful preparation of the surface of the specimens under test. For example, by taking specimens of alloy C phosphor bronze, 8 numbers hard, in the "as received" condition and subjecting them to a microscopic polish prior to testing, it is possible to raise the endurance limit from 26,000 lb. per sq. in. to 28,500 lb. per sq. in.

The data in Table II and the curves of Fig. 6 show that plated finishes may, in the absence of proper surface preparation, materially lower the fatigue resistance of non-ferrous metals. The tests made on phosphor-bronze specimens to which various nickel, chromium, and nickel-chromium finishes have been applied illustrate this. The data also show that part of this loss may be overcome by increasing the adherence of the plated finishes by sand-blasting or other suitable surface preparation. Likewise, holes, notches or formed offsets in highly stressed sections will reduce the fatigue properties below that normally expected of a given material as shown by the data for alloy B nickel silver sheet, 6 numbers hard.

Improvement in the fatigue properties of non-ferrous sheet metals may be obtained by proper alloying. This is shown by comparing the curves presented in Fig. 7 with the endurance limit for sheet copper. By the addition of beryllium and a small percentage of nickel to copper, it is possible to raise the endurance limit from 14,000 lb. per sq. in. for copper sheet 6 numbers hard to 36,000 lb. per sq. in. for beryllium-copper in the optimum cold-worked and heat-treated condition.

Improvement in the fatigue characteristics of non-ferrous sheet may also be obtained through refinement or control of grain size. This is illustrated by the curves in Fig. 8 where it is shown that alloy B nickel silver having a grain size of 0.017 mm. has better fatigue characteristics than nickel silver of similar composition and hardness but of larger grain size.

With moderate amounts of cold work, nickel silver sheet has better fatigue characteristics when specimens are blanked at 45 deg. than parallel or at 90 deg. to the direction of rolling as shown by the curves of Fig. 9.

Alpha brass hardened by the addition of nickel-silicide shows some improvement in the endurance limit but more complete test data indicate that the improvement in the ratio of endurance limit to tensile strength reported in the previous paper[2] is not borne out. More complete fatigue data show but a slight gain in fatigue properties with decided improvement in tensile properties. The gain in fatigue properties is not considered sufficient to justify the use of these alloys. However, dispersion-hardened alloys of the beryllium-copper type do have superior fatigue properties to the dispersion-hardened alpha brasses and are comparable in their minimum fatigue characteristics to cold-worked alloy C phosphor bronze. It is possible by optimum cold working and aging of beryllium-copper alloys to obtain somewhat higher fatigue properties but these results are not obtained consistently.

CONCLUSIONS

The test results indicate that the fatigue endurance limit at 100,000,000 reversals of stress varies from approximately 13.5 to 40 per cent of the tensile strength. High tensile strength alone does not necessarily imply good fatigue characteristics.

The fatigue characteristics of non-ferrous sheet metals may be raised by cold working but the resulting increase is not proportional to the increase in tensile strength.

Precipitation hardening of alpha brass by the addition of nickel silicide increases the endurance limit.

Grain direction in materials such as alloy B nickel silver and cold-worked aluminum alloy No. 17S affects the endurance properties but in the cases of alloys such as 2S, 3S, 17S-O and 17S-T where the microstructure reveals little or no directional properties the fatigue properties are likewise unaffected by grain direction.

Small holes or additional working of highly stressed sections reduces the endurance limit below that of the original material. By proper surface preparation such as polishing the endurance limit may be materially raised.

The endurance limit will be lowered by the addition of plated finishes unless proper precautions are taken to prepare suitably the surface prior to the application of the plated finish.

By the use of material having a small grain size, improvement in the endurance properties may be obtained.

Alloys such as beryllium-copper, aluminum alloy 17S and K Monel as well as the dispersion-hardened brasses may be heat treated to obtain improved fatigue characteristics.

Acknowledgments:

The authors are indebted to Messrs. J. P. Guerard and A. H. Falk who assisted in the laboratory work. Many helpful suggestions and criticisms have also been received from Mr. J. R. Townsend, in whose department this work was conducted.

DISCUSSION

Mr. H. L. Burghoff[1] (*presented in written form*).—The authors are to be commended for the presentation of such a fund of endurance data, which are, I believe, the most comprehensive yet published on non-ferrous materials.

The authors' findings on the effects of grain size and of direction of rolling with respect to length of specimen are interesting. We are now conducting a series of tests on brasses, using similar machines and specimens, and considering variations in temper and directional properties. While our tests are not yet complete, indication is given that material with small grain size has higher endurance limit than similar material with large grain size, which is in agreement with the authors' results on nickel silver. With regard to directional properties of rolled brass, our present results tend to indicate that specimens cut at 90 deg. to the rolling direction have higher endurance limit than specimens cut at 45 deg., which in turn are superior to those cut at 0 deg. or parallel to the rolling direction. This agrees with the authors' results on nickel silver rolled 10 B & S numbers hard, but not as rolled 4 B & S numbers hard.

Results are given in Table II of the paper showing that the effect of a low temperature anneal, actually a stress-relieving anneal, is to increase the endurance limit of rolled nickel-silver specimens cut parallel to the rolling direction. Again referring to our work with brass, we are obtaining comparable results with specimens similarly cut and subjected to a stress-relieving anneal. The effect of this anneal on specimens cut at 45 and 90 deg. to the rolling direction is less, however, decreasing as the angle increases. It is hoped to embody our work in a suitable paper, upon its completion.

In making use of endurance limit values for design purposes, consideration must often be given to modulus of elasticity. Thus, two materials having the same endurance limit but different moduli of elasticity might be under consideration. If the application were one in which a certain deflection were to be made, regardless of stress set-up, the logical choice would be the material of lower modulus, in which the stress would be less. This would be a different problem from an application in which a definite load is to be carried. To assist in the evaluation of various materials tested, it is our practice to report the actual deflection of specimens corresponding to the endurance limit. This makes for ready comparisons, being, of course, a further indication of elastic modulus which is desirable because of the varying values of modulus reported for non-ferrous alloys. In the present paper, steps in this direction have been taken, values of moduli of elasticity being given in Table II and correlation of stresses and deflections being given in Table III.

Mr. C. H. Davis[2] (*presented in written form*).—A few words may be in

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[1] Research Metallurgist, Chase Brass and Copper Co., Waterbury, Conn.

[2] Metallurgist, The American Brass Co., Waterbury, Conn.

order with regard to the heat hardenable alloys, such as beryllium-copper. A maximum endurance limit of 36,000 lb. per sq. in. is given for a specimen 4 numbers hard, aged 2 hr. at 275 C. If the specimens 2 numbers hard had been aged for a longer time, or at a slightly higher temperature, their endurance limit would undoubtedly have been higher. The same comment applies to the annealed specimens.

A point worthy of discussion, but which has been omitted with quite good propriety from the paper, is that of aging after forming. An example follows:

Phosphor bronze, grade C annealed, has an endurance of 21,000 lb. per sq. in., but when rolled 8 or 10 numbers a limit of 27,000 lb. per sq. in., and when in such a hard condition the bronze could not be bent or formed to any appreciable degree. A piece of beryllium-copper, on the other hand, may be formed and even drawn in the annealed condition, then given a heat treatment or aging, which would yield an endurance limit of 28,000 to 31,000 lb. per sq. in. Moreover, the beryllium-copper thus treated would have excellent spring properties.

Taking this view, the comparison would be between the phosphor bronze with a limit of 21,000 lb. per sq. in. and beryllium-copper with an endurance limit of 28,000 to 31,000 lb. per sq. in. Actually, in commerce, many thousands of pounds of this heat-hardenable alloy are being used because of this very ability to become hardened and strengthened after the final annealing and forming operations. With the advances in the art of manufacturing beryllium-copper, since the time the specimens reported in the paper were prepared, the properties obtained after heat treatment are fairly uniform. The alloy is sensitive to minor variations in heat treatment but with this knowledge the properties can be improved by exact selection of proper conditions for aging.

MR. R. L. TEMPLIN.[3]—The authors are to be complimented on doing an excellent piece of work in evaluating the fatigue properties on some of the nonferrous metals. Their findings in connection with effects of grain size on the fatigue properties are quite in accordance with those previously reported before this Society[4] for some of the aluminum alloys. They find also, as was previously reported for aluminum alloys, that the fatigue strength increases with the cold working but at a much lower rate than the static mechanical properties. The beneficial effects of strain relief have been recognized generally, at least in a qualitative way, but it is gratifying to obtain quantitative data for specific materials. The improvements attributable to strain relief, in so far as fatigue properties are concerned, will of necessity depend upon the amount of internal strain in the material, initially, as well as upon the metal itself.

The effects noted with respect to the direction of rolling are not entirely in accordance throughout the data given. It would seem reasonable to expect differences, depending not only on the amount of rolling but the details of the fabrication process used; that is, the amount of rolling in a given direction as well as the rate of reduction will undoubtedly have some effect on the resulting fatigue properties.

MR. E. E. THUM.[5]—I should like to ask Mr. Templin's opinion as to the scatter of the test results on the age-hardening alloys. Is it his thought that the

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[3] Chief Engineer of Tests, Aluminum Company of America, New Kensington, Pa.

[4] R. L. Templin, "The Fatigue Properties of Light Metals and Alloys", *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 364 (1933).

[5] Editor, *Metal Progress*, American Society for Metals, Cleveland, Ohio.

scatter shown in this paper is an evidence that another heat treatment is required, or should we expect a scatter as being inherent to that type of metallurgical product?

Mr. Templin.—Answering Mr. Thum's question, I must confess an unfamiliarity with the age-hardening beryllium-copper alloys. In our early efforts to produce age-hardening aluminum alloys we sometimes got an appreciable scatter in the fatigue values similar to that referred to, but in general, such scatter of values is absent in our fatigue tests of present-day aluminum products. Occasionally in certain forms of products we note such a scatter but whether or not it can be attributable to age-hardening or other factors, it is rather difficult to say.

Mr. G. R. Gohn.[6]—We are pleased to have the written comments by Messrs. Burghoff and Davis on the paper by Mr. Greenall and myself. We are particularly glad to find that other investigators are in agreement with our findings with respect to the effect of grain size and low temperature heat treatments on fatigue strength. Mr. Burghoff, working on brass, reports the same effect with respect to grain size and low-temperature anneals as we have found on nickel-silver sheet. Mr. Burghoff also reported somewhat better fatigue properties for brass specimens taken at 90 deg. than for specimens taken parallel to or at 45 deg. to the direction of rolling. In our paper we have reported the effects of grain direction on 11 alloys, 5 of which were aluminum. In these particular aluminum alloys we observed no marked directional effect, but in the other alloys, particularly the nickel-silver sheet which we use for springs, we did find some improvement at 45 deg. This evidence is, however, not sufficient for us to state that all alloys will give the same effect.

In Mr. Davis' comments he stated that, as information becomes available and methods of manufacture improve, undoubtedly better fatigue properties will be obtained for beryllium-copper sheet. This is borne out by the last comment of Mr. Templin who, in reply to Mr. Thum's question, stated that heat-treated aluminum alloys which have been manufactured commercially for a considerable period of time, at present show very little scatter in the data obtained in fatigue tests on those alloys. We believe that as the methods of manufacture and heat treatment improve, both for heat-treated nickel and copper alloys, the scatter obtained in fatigue tests on these alloys will likewise be decreased, and that the fatigue properties will be more uniform than those which we have noted. We should like to point out that the heat treatments which we employed were those recommended by manufacturers of these alloys, and in many cases the alloys themselves were heat treated by the manufacturer and not by us.

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[6] Member of Technical Staff, Bell Telephone Laboratories, Inc., New York City.

FATIGUE PROPERTIES OF METALS USED IN AIRCRAFT CONSTRUCTION AT 3450 AND 10,600 CYCLES

By T. T. Oberg[1] and J. B. Johnson[1]

Synopsis

A description of a ball-bearing rotating-beam machine operating at 10,600 cycles and the results of comparative tests on these machines and ball-bearing and plain-bearing machines operating at 3450 and 1725 r.p.m., respectively. Plain and notched specimens were used and operating temperatures discussed. The values for the fatigue limits were not appreciably affected by this range of speed.

Several investigators have reported fatigue tests made at frequencies above the normal operating frequency for standard rotating-beam machines of 1700 to 3500 cycles per minute. Jenkin and Lehmann[2] used a flat specimen driven by an air blast and made tests at frequencies from 36,000 to 1,200,000 cycles per minute. They reported an increase of approximately 20 per cent in the fatigue limit at the highest frequency but the extrapolated curves show very slight increase below 30,000 cycles. The National Bureau of Standards[3] with a similar machine reported tests made at 12,000 cycles per minute for sheet-metal specimens which are comparable with results on rotating-beam specimens at 3450 cycles per minute. Krouse[4] described a rotating cantilever machine and indicated a higher fatigue limit at 10,000 cycles for brass and stainless steel, but no change in several other metals. All of the metals had a higher fatigue limit at 30,000 than at 15,000 cycles. Boone and Wishart[5] submitted results on a similar machine but no comparative data on effect of speed were given.

A large amount of data has been assembled on the fatigue properties of aircraft materials using the R. R. Moore plain-bearing rotating-beam type machine at operating speeds of 1725 and 3450 cycles per minute. The desirability of developing a similar machine which would operate at higher speeds and accommodate the same type of specimen is obvious. The latest model of such a machine, Fig. 1, consists of two cast aluminum housings in which are mounted ball bearings supporting the nitrided steel spindles in which the specimens are held. The spindles are driven through a rubber-encased splined connection

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[1] Materials Testing Engineer, Air Corps, and Chief, Material Branch, respectively, Wright Field, Dayton, Ohio.

[2] C. F. Jenkin and G. D. Lehmann, "High Frequency Fatigue," *Proceedings*, Royal Soc. (London), Vol. 125-A, (1929).

[3] Reports of National Advisory Committee for Aeronautics, 1930 and 1931, U. S. Government Printing Office, Washington, D. C.

[4] G. N. Krouse, "A High-Speed Fatigue Testing Machine and Some Tests of Speed Effect on Endurance Limit," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 156 (1934).

[5] W. D. Boone and H. B. Wishart, "High-Speed Fatigue Tests of Several Ferrous and Non-Ferrous Metals, at Low Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part II, p. 147 (1935).

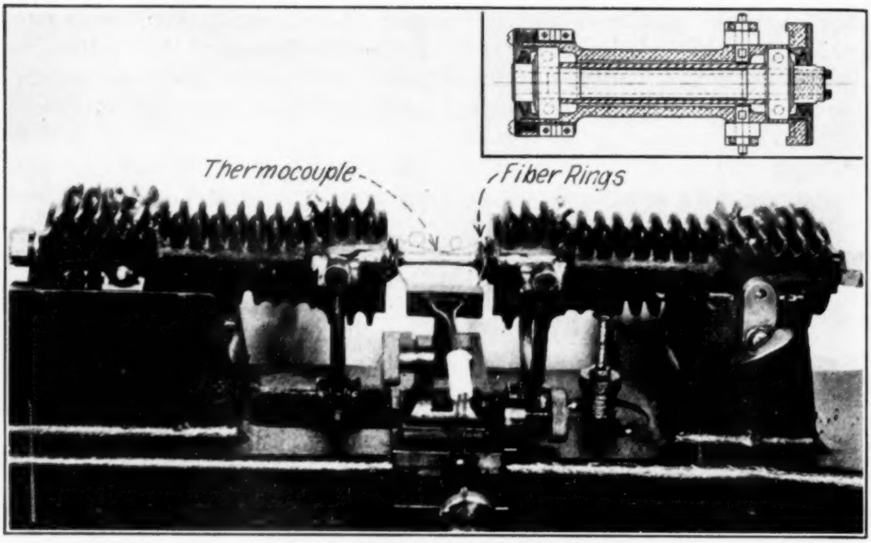

FIG. 1.—Fatigue Testing Machine.

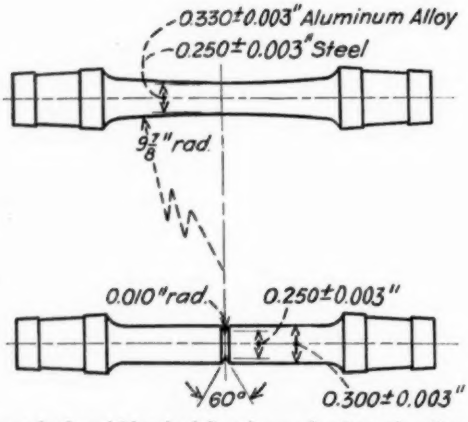

FIG. 2.—Unnotched and Notched Specimens for Rotating-Beam Machine.

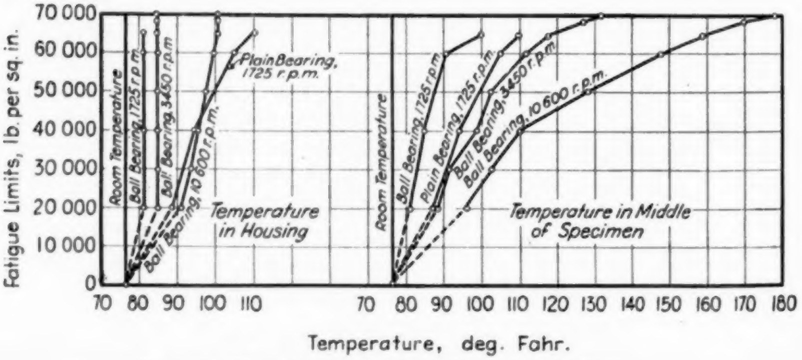

FIG. 3.—Chromium Nickel (18–8) Steel, Cold Drawn.

TABLE I.—CHEMICAL ANALYSES AND PHYSICAL PROPERTIES OF MATERIALS TESTED.
¾-in. round bars.

Material	Condition	Chemical Composition, per cent							Tensile Properties					
		Carbon	Manganese	Nickel	Chromium	Molybdenum	Iron		Tensile Strength, lb. per sq. in.	Yield Strength, lb. per sq. in.		Elongation in 2 in., per cent	Reduction of Area, per cent	Brinell Hardness, 3000-kg. load
										Set 0.002 in.	Set 0.00005 in.			
S.A.E. No. 2330	Quenched in oil, 1500 F. Drawn at 1100 F., 30 min.	0.29	0.52	3.37	......	......	......		128 700	127 000	117 000	20	67	257
S.A.E. No. 4130	Normalized	0.31	0.65	......	0.71	0.17	......		76 200	45 000	30 500	32	66	145
	Quenched in oil, 1625 F., Drawn at 1100 F., 30 min.	0.31	0.65	......	0.71	0.17	......		139 500	120 000	90 500	18	63	290
	Quenched in oil, 1625 F., Drawn at 650 F., 30 min.	0.31	0.65	......	0.71	0.17	......		199 300	168 500	100 000	10	45	432
Chromium-nickel	Annealed	0.06	0.45	9.98	19.98	......	......		82 800	34 800	15 000	64	76	150
	Cold drawn	0.05	......	8.87	20.09	......	......		132 500	109 000	42 000	32	72	277
Inconel	Annealed	0.04	0.12	80.9	13.6	......	5.7		101 600	44 300	18 000	40	67	178
	Cold Drawn	0.04	0.12	80.9	13.6	......	5.7		178 500	172 000	101 000	7	27	340
		Copper	Silicon	Iron	Manganese	Zinc	Tin	Aluminum						(500-kg. load)
Alcoa 27S-T, 6-in. forging	Quenched in water 950 F., 12 hr., Aged at 370 F., 18 hr.	4.18	0.83	0.35	1.10	......	0.05	Remainder	60 000	47 000	35 000	12	24	120
Alcoa X-164,[a] casting	Quenched in water 960 F., 16 hr., Aged at 320 F., 6 hr.	5.36	0.11	0.49	0.52	2.23	......	Remainder	51 000	42 000	......	3	......	116

[a] Experimental alloy developed by the Aluminum Company of America.

directly connected to a $\frac{1}{4}$-hp. motor operating at 10,600 r.p.m. The ball-bearing machines have lower starting and running torques, and the housings operate at lower temperatures, Fig. 3, than the plain-bearing machines. No difficulty has been experienced with the lubrication of the machines, a light oil having a viscosity at 100 F. of 80 to 115 sec. has proved satisfactory. A few drops of fresh oil are added at about 48-hr. intervals. All tests except those of the Alcoa 27S-T at 3450 r.p.m. and the tests at 1725 r.p.m. were made in ball-bearing machines.

TABLE II.—FATIGUE PROPERTIES OF METALS AT AN OPERATING SPEED RANGING FROM 1725 TO 10,600 R.P.M.

Material	Condition	Fatigue Limit Rotating Beam, lb. per sq. in.					
		Unnotched			Notched		
		Plain Bearing, 1725 r.p.m.	Ball Bearing, 3450 r.p.m.	Ball Bearing, 10,600 r.p.m.	Plain Bearing, 1725 r.p.m.	Ball Bearing, 3450 r.p.m.	Ball Bearing, 10,600 r.p.m.
S.A.E. No. 2330	Drawn at 1100 F.	79 000	80 000	79 000	31 000	....	31 000
S.A.E. No. 4130	Normalized	....	42 000	43 000	....	25 000	25 000
S.A.E. No. 4130	Drawn at 1100 F.	....	86 000	86 000	....	30 000	30 000
S.A.E. No. 4130	Drawn at 650 F.	114 000	....	114 000	....	53 000	53 000
Chromium-nickel	Annealed	....	36 000	35 000	....	43 000	42 000
Chromium-nickel	Cold drawn	....	70 000	70 000	48 000	....	50 000
Inconel	Annealed	....	47 000[a]	50 000[a]	....	....	....
Inconel	Cold drawn	....	53 000[b]	56 000[b]	....	....	....
Alcoa 27S-T., 6-in forging	Heat treated	....	11 000[d]	15 000[c]	....	6 000[b, d]	7 000[b]
Alcoa X-164, casting	Heat treated	....	10 000[b]	10 000[b]	....	....	....

[a] Fatigue limit at 100,000,000 cycles.
[b] Fatigue limit at 200,000,000 cycles.
[c] Fatigue limit at 500,000,000 cycles.
[d] Plain-bearing machine.

Test Procedure:

The *S-N* diagrams for the fatigue properties were obtained in the usual manner by running the specimens to failure, except in the case of the aluminum alloy, Fig. 11, in which case the specimens which had been tested for 300,000,000 to 2,700,000,000 cycles without failure were retested at a higher stress. The procedure was simply to apply the additional load without stopping the machine and continue the test until the specimen failed.

The specimens, Fig. 2, were the short type identical to those used with the plain-bearing machines. The unnotched specimens were rough machined $\frac{1}{8}$ in. oversize, heat treated, ground to size, and polished longitudinally with Manning No. 0, 00 and 000 papers. The notched specimens were machined and the notch cut with a special tool made by Pratt & Whitney Co. The depth and root diameter were measured on a Zeiss toolmaker's microscope.

Material:

Two representative aircraft steels of low alloy content, S.A.E. No. 2330 and No. 4130, and one of high-alloy content, 18 per cent chromium, 8 per cent nickel; a non-ferrous nickel-base alloy, Inconel; and two aluminum-base heat-treated alloys were tested. All of the specimens were machined from rolled or forged bars except one of the aluminum alloys which was in the form of test bars cast to shape in a green-sand mold. The skin was removed and the specimens polished. The chemical composition, heat treatment, and tensile properties are given in Table I.

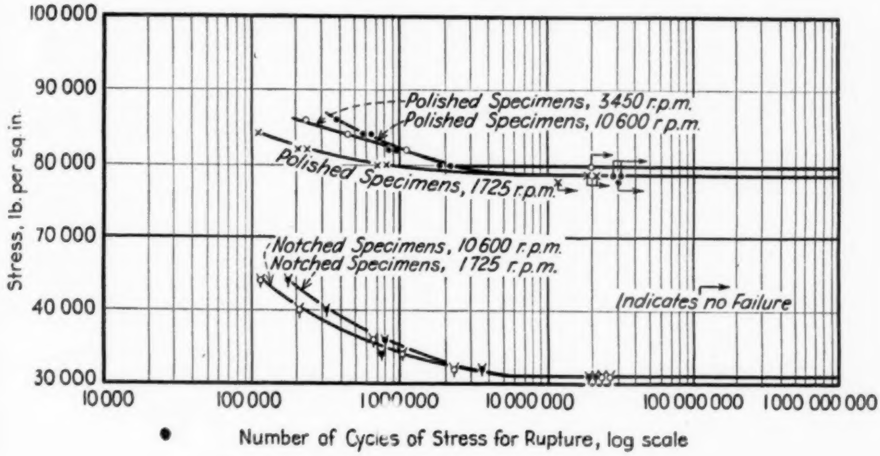

FIG. 4.—S.A.E. No. 2330 Steel Bar, Heat Treated.

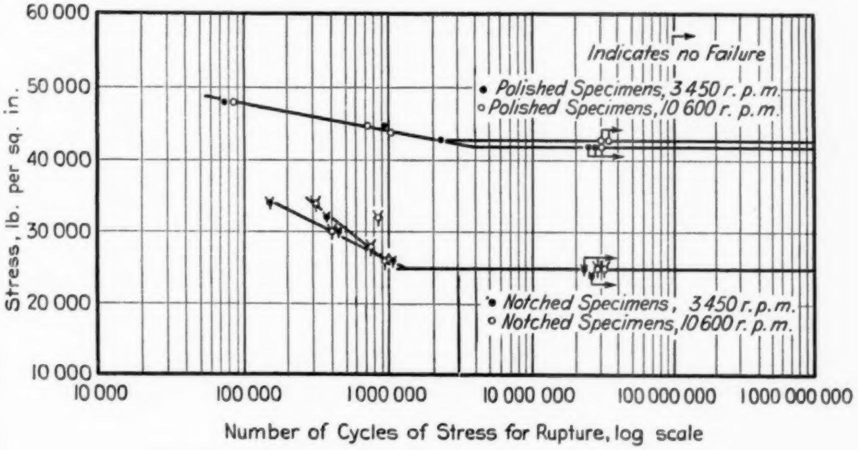

FIG. 5.—S.A.E. No. 4130 Steel, Normalized and Annealed.

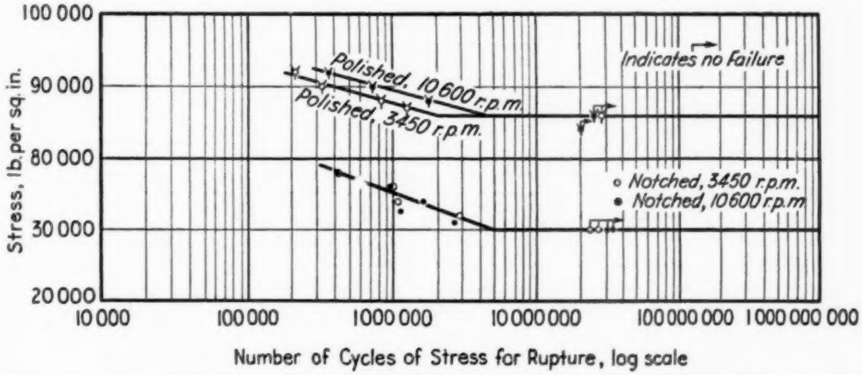

FIG. 6.—S.A.E. No. 4130 Steel, Oil Quenched at 1625 F., Drawn at 1100 F.

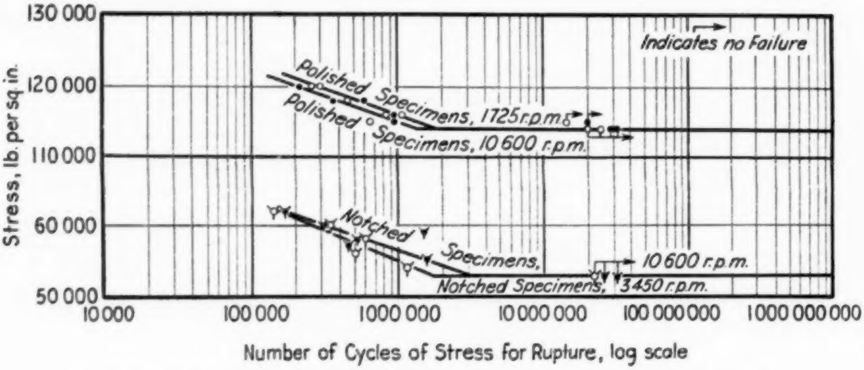

FIG. 7.—S.A.E. No. 4130 Steel, Oil Quenched at 1625 F., Drawn at 650 F.

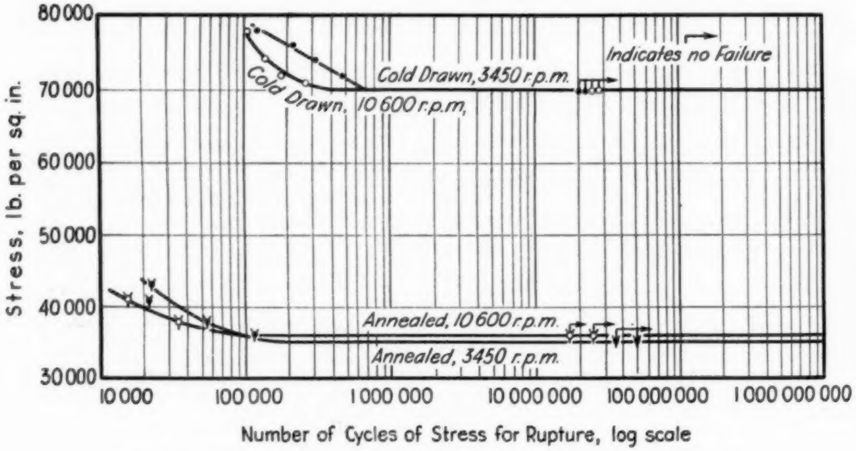

FIG. 8.—Chromium Nickel (18-8) Steel Bars, Polished Specimens.

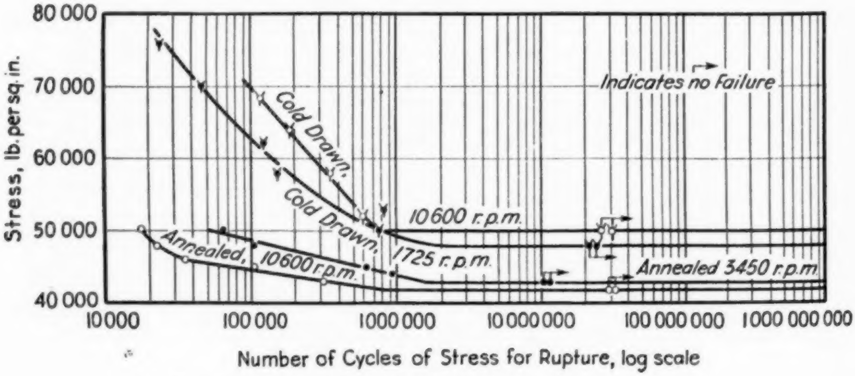

FIG. 9.—Chromium Nickel (18-8), Notched Specimens.

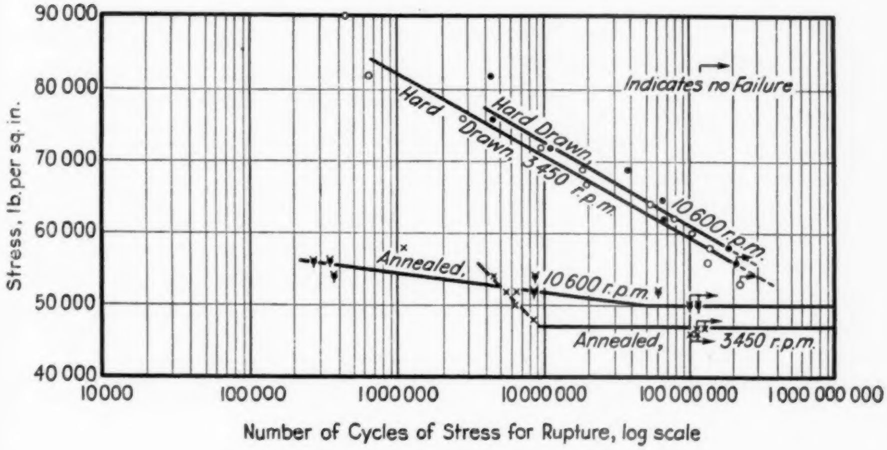

FIG. 10.—Inconel, Polished Specimens.

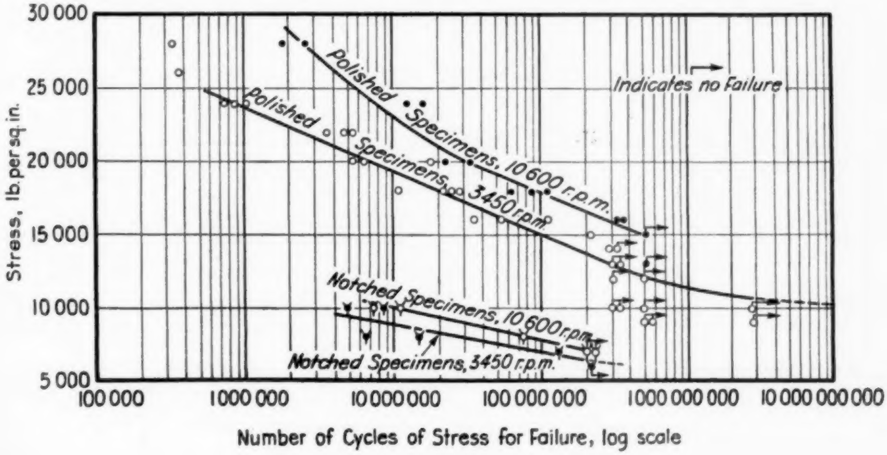

FIG. 11.—Forged Aluminum Alloy.

ORIGINAL TESTS		RETESTS	
STRESS, LB. PER SQ. IN.	NUMBER OF CYCLES TO RELOAD	STRESS, LB. PER SQ. IN.	NUMBER OF CYCLES TO FAILURE
POLISHED SPECIMENS, 10,600 R.P.M.			
15 000	502 560 000	20 000	660 650
13 000	498 500 000	20 000	2 780 000
POLISHED SPECIMENS, 3450 R.P.M.			
14 000	300 150 000	18 000	2 425 000
13 000	302 705 000	18 000	28 078 000
13 000	310 560 000	18 000	432 000
12 000	308 535 000	18 000	1 858 000
12 000	500 500 000	18 000	4 408 500
10 000	302 635 000	18 000	21 353 300
10 000	302 134 000	18 000	9 905 000
10 000	503 450 000	18 000	8 860 000
10 000	2 720 500 000	18 000	33 045 000
9 000	503 000 000	18 000	24 627 000
9 000	506 450 000	18 000	38 383 000
9 000	2 725 150 000	18 000	70 250 000

Results of Tests:

The *S-N* diagrams are shown in Figs. 4 to 12 and Table II. It is apparent that for the ball-bearing machines operating at 3450 and 10,600 r.p.m. the diagrams are practically superimposed for both the notched and unnotched specimens, with the exception of Inconel, Fig. 10, and 27S, Fig. 11. The slight differences in fatigue limit although recorded in the table are of doubtful significance. For stresses above the fatigue limit the diagrams diverge in some cases, but in this region it is seldom that exactly the same life will be obtained on two identical specimens when the tests are made at the same frequency. Results obtained in different laboratories or in the same laboratory on different lots of specimens often indicate marked variability.

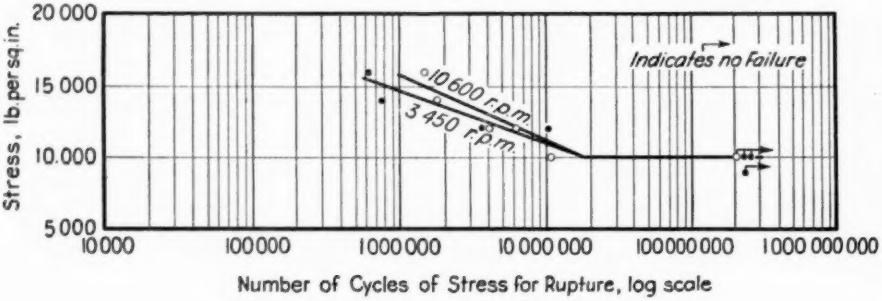

FIG. 12.—Cast Aluminum Alloy, Polished Specimens.

The diagrams for the austenitic chromium-nickel steel in the annealed condition indicate the unusual phenomenon, which has been noticed by other investigators, that the notched specimens have a higher fatigue limit than the unnotched specimens. The stresses are calculated from the formula for beams of uniform section which contains no stress concentration factor, but the results are comparable with those for other wrought structural metals which invariably show a lower fatigue limit for notched specimens.

Moore and Kommers[6] state that "when metal is stressed within the fatigue range, the cyclic state is attained by plastic deformation or strain hardening. The cyclic state is attained when plastic strain ceases, and the metal can then withstand the cycle of stress indefinitely."

The fatigue limit of annealed chromium-nickel austenitic steel is above the tensile yield strength of the material and in the range where plastic strain is large. Plastic strain is influenced by the shape of the specimen. The deflections for equal loads were greater for the unnotched than for the notched specimens; therefore, plastic strain will cease at a greater load in the notched than in the unnotched specimens and the fatigue limit will be correspondingly higher. The higher fatigue strengths as shown in the *S-N* diagrams for alloy 27S-T, Fig. 11, at 10,600 cycles are probably not due entirely to the greater frequency. The specimens for both diagrams were machined from the same bar and at approximately the same location relative to the outside surface, but the tests at 10,600 cycles were made more than a year later than those at 3450 r.p.m. The fatigue properties may have improved through aging although the tensile properties were not measurably affected. The

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[6] H. F. Moore and J. B. Kommers, "The Fatigue of Metals," p. 47, McGraw-Hill Book Co., Inc., New York City (1927).

tests at 3450 r.p.m. were made in plain-bearing machines which introduce a small shear load in the specimen. The tests at 10,600 r.p.m. were not carried beyond 500,000,000 cycles, but the two specimens which had run for this length of time and were retested at 20,000 lb. per sq. in. showed damage. The fatigue limit at 10,600 r.p.m. is less than 13,000 lb. per sq. in. and therefore is close to that determined for 3450 r.p.m. The lower curve indicates that the fatigue limit for this alloy when tested at 3450 r.p.m. is above 10,000 and below 12,000 lb. per sq. in. The two specimens run for 2,700,000,000 cycles and 9000 and 10,000 lb. per sq. in. and retested at 18,000 lb. per sq. in., not only indicated no damage but a higher fatigue strength than specimens originally tested at 18,000 lb. per sq. in. Since it took 18 months to complete the tests at the lower stresses, the specimens at retest were actually the same age as the specimens tested at 10,600 cycles.

Some scatter in the retested specimens is noticeable. This is characteristic of all fatigue tests on aluminum alloys containing a hardening constituent present in the form of small particles. It is reasonable to believe that when one of these particles is lodged at the most highly stressed fiber it will gradually loosen and act as a stress raiser causing erratic behavior.

All aluminum alloy specimens were coated with a thin film of petrolatum immediately after machining and occasionally during the test. A microscopic examination of the surfaces of the specimens running for long periods indicated no pitting or corrosion.

The heat developed at the stressed section of a rotating specimen depends upon the chemical composition and physical condition of the material but is higher at a frequency of 10,600 than at 3450 cycles. The differences in temperatures increase with increase in the applied stress, Fig. 3. These curves are typical, for annealed materials the temperatures are higher and for quenched-and-drawn structural steels somewhat lower. Temperatures were measured by a chromel-alumel thermocouple soldered to the middle of the specimen, Fig. 1. The thermocouple wires were looped to give flexibility and soldered to rings of similar material mounted on cylindrical fiber blocks attached to the shoulders of the specimen. The potentiometer leads in the pivoted support permitted quick and positive contact with the rings when the machine was stopped. The specimen was run until the temperature had come to equilibrium. Several readings for each load were taken by stopping the machine at 10-min. intervals. The temperatures drop very rapidly after stopping the machine indicating that only a small section attains the maximum temperature. Several organic compounds with known melting points were applied to the specimens. The temperatures indicated by this method checked the potentiometer readings very closely.

Conclusions:

The fatigue limits determined on ball-bearing machines operating at 10,600 r.p.m. are comparable with those obtained on ball-bearing machines or plain-bearing machines operating at a lower number of cycles. The shape of the *S-N* diagram may be affected at stresses above the fatigue limit. This was not established by these tests.

The temperature rise in the section of the specimen subjected to the maximum stress increases with an increase in frequency, but the fatigue limit is not affected.

The forged aluminum alloy 27S-T has a fatigue limit which has been established as satisfactorily as the fatigue limits of other metals.

DISCUSSION

Mr. R. L. Templin.[1]—The authors of this paper have used one of the more common types of notches, but the results obtained with such a notch obviously do not show what reductions in fatigue limits may be expected when other forms of notches are used. The form and size of notch used are quite similar to those of the notch we have used in the Aluminum Research Laboratories, as reported previously before this Society.[2] In our own notch-fatigue tests we do not expect to get quantitative values for design purposes but use the results as criteria of the notch sensitivity of the various grades and tempers of aluminum alloys. If the results are used in this way rather than quantitatively, as many designers would like to use them, they serve a very useful purpose. It is very difficult to select quantitative values from laboratory fatigue tests, which can be used as design values in actual engineering structures.

I should like to ask the authors if they have made any high-speed fatigue tests on alloys which have fatigue limits appreciably above their proportional limits. Apparently no such data are given in the paper.

Mr. Rudolf K. Bernhard.[3]—We made tests similar to those described by Messrs. Oberg and Johnson, rebuilding completely the old R. R. Moore fatigue machine in order to obtain higher speeds and to shorten the time for testing. First of all, the important question to be decided was whether these higher speeds have any influence on the results. The highest frequency obtained was 15,000 r.p.m. The standard high-speed R. R. Moore fatigue machine now has a range of from 1000 to 10,000 r.p.m. I made a considerable number of tests with specimens of various carbon steels and different types of brass—both plain and notched specimens—and could find no substantial difference in the fatigue limit for the speed range up to 10,000 r.p.m. These results coincide very well with the statements made by Messrs. Oberg and Johnson.

Mr. H. B. Wishart[4] (*by letter*).—The question most frequently arising when high-speed fatigue testing is mentioned is: What effect does the increased speed have on the endurance limit of metals? The authors present data showing that for speeds of testing up to 10,600 r.p.m. no appreciable change was noted in the endurance limit of the metals reported.

The writer has made a number of tests on high-speed fatigue machines and on one or two occasions has found that the endurance limit as determined at 10,000 r.p.m. and 3600 r.p.m. has varied as much as 10 per cent. There are some experimenters who would condemn the high-speed machine for this variation. However, when all points are considered it is to be remembered that there are at present no standards for making fatigue tests. Results obtained from the high-speed machine could be considered standard tests as much as the results

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[1] Chief Engineer of Tests, Aluminum Company of America, New Kensington, Pa.

[2] R. L. Templin, "The Fatigue Properties of Light Metals and Alloys", *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 364 (1933).

[3] Consulting Engineer, Baldwin-Southwark Corp., Philadelphia, Pa.

[4] Metallurgist, Carnegie-Illinois Steel Corp., Gary Works, Gary, Ind.

from the low-speed machine, the advantage of the high-speed machine being that an endurance limit based on 10,000,000 cycles can be determined for most ferrous metals in one-fourth the time of the low-speed machine.

The chief objection to the use of fatigue testing in industry is the slowness of the test when speeds of 1750 or 3450 r.p.m. are used. Therefore, every means should be used in order to increase the speed of making fatigue tests within the limits of obtaining consistent and comparative results. The authors have made a good start in this direction and are to be complimented on their development of a machine for this purpose.

MESSRS. T. T. OBERG[5] AND J. B. JOHNSON[5] (*authors' closure, by letter*).—The authors do not recommend using the results of notch-sensitivity tests, quantitatively, for design purposes, but have found that the notch used in this investigation gives a fair measure of the notch-sensitivity of material, that is, the resistance to failure from fatigue when stress raisers are present at the surface of the part.

The corrosion-resistant steel and Inconel, Tables I and II, have fatigue limits above their proportional limits (set = 0.00005 in.)

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[5] Materials Testing Engineer, Air Corps, and Chief, Material Branch, respectively, Wright Field, Dayton, Ohio.

A FATIGUE MACHINE FOR TESTING METALS AT ELEVATED TEMPERATURES

By F. M. Howell[1] and E. S. Howarth[1]

Synopsis

This paper describes and discusses in some detail a fatigue machine for testing metals at elevated temperatures. The specimen used is part of a cantilever beam, one end of which is fixed in the electrically heated furnace. While the specimen is entirely within the furnace, the deflected beam assembly extends outside the furnace wall and is revolved in a circle by a variable eccentric driven at 3600 r.p.m. A unique calibration apparatus is used to load the specimen and to measure the resulting deflection while the machine is in operation. From the load-deflection relationship thus obtained, the stress in the test specimen can be calculated.

The method of temperature control is described. Specimen temperatures from room temperature to 600 F. can be controlled within ±2 deg. Fahr.

Errors due to speed effect, misalignment, variation in specimen size and method of stress calculation are discussed. These errors indicate that the maximum stress in the specimen can be calculated within ±3 per cent.

Calibration curves and results of fatigue tests on one of the commercial aluminum alloys representative of the type of data obtainable are included.

Considerable tension- and creep-test data have been obtained on aluminum alloys at elevated temperatures, but largely because of inadequate testing equipment little reliable work has been done to determine the effect of elevated temperatures on the fatigue properties of these alloys. One of the recent additions to the equipment of the Aluminum Research Laboratories has been four high-temperature fatigue machines which are being used at present to investigate the fatigue properties of aluminum forging and casting alloys employed primarily in aircraft-engine construction. The machines are, however, suitable for testing other metals and alloys.

It is the purpose of this paper to describe and discuss these testing machines and the auxiliary calibration apparatus used in running the fatigue tests at elevated temperatures. The data and results for one wrought aluminum alloy, Alcoa 17S–T, are presented merely to demonstrate the method of stress calculation and to indicate the type of data obtainable.

Description of the Test Units

The high-temperature fatigue machines here discussed were developed from a preliminary experimental unit of the rotating-beam type which has been previously described.[2] The preliminary

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[1] Engineer of Tests, and Research Engineer, respectively, Aluminum Research Laboratories, Aluminum Company of America, New Kensington, Pa.

[2] R. L. Templin, "The Fatigue Properties of Light Metals and Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 364 (1933).

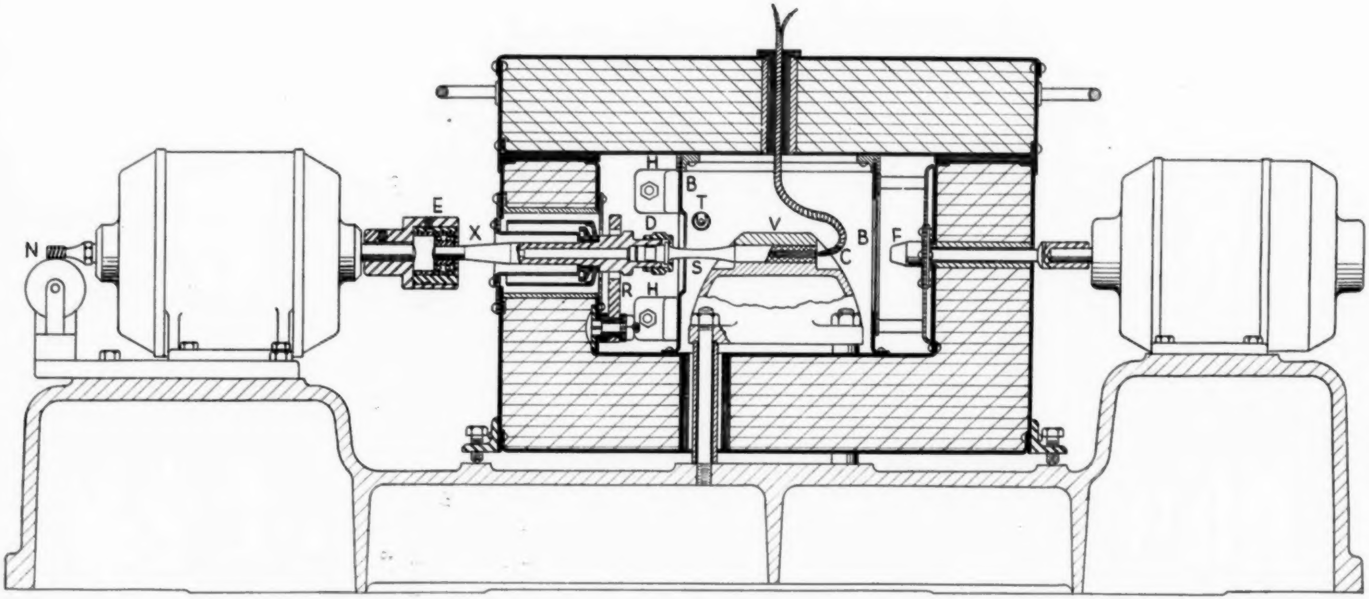

FIG. 1.—High-Temperature Fatigue Machine of a Fixed Cantilever-Beam Type.

V—vise
B—baffle box
F—fan

S—specimen
X—extension
H—strip heaters

T—thermoregulator
R—stop ring
C—thermocouple

E—variable eccentric
N—counter
D—connecting nut

unit was unsuccessful because the bearings which were contained within the furnace did not operate satisfactorily at the required temperatures. From this experimental unit, however, there was developed the fixed cantilever-beam type machine with all bearings outside the furnace so that no bearings are required to operate at or near the temperature of the specimen. A machine of this general type has been used in the Fatigue of Metals Laboratory of the University of Illinois for tests at room and elevated temperatures.[3] While the specimen is entirely within the furnace, the deflected end of the cantilever-beam assembly extends outside the furnace wall and is revolved in a circle by a variable eccentric driven at 3600 r.p.m.

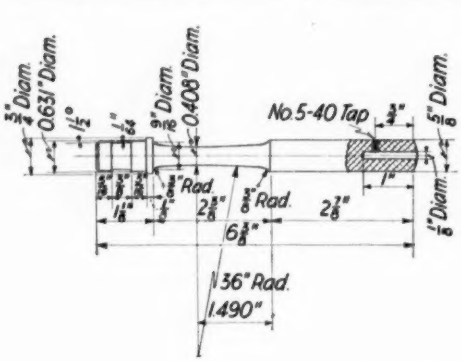

FIG. 2.—Details of Specimens Used in High-Temperature Fatigue Machines.

The general drawing of one of the units is shown in Fig. 1. In the center of the well-insulated furnace box is the rigid specimen vise *V* which is fastened to the cast iron base by supporting legs which extend through the bottom of the furnace. Surrounding the vise is the sheet-metal baffle box *B* which has a hole in the right end at the circulating fan *F* and another in the left end through which the cantilever beam passes. The air is drawn from the inside of the baffle box by the fan and is passed over the electric strip heaters *H*, located on the front and rear furnace walls, to the left end where it reenters the baffle box, passing around the specimen *S* which is clamped rigidly in the specimen vise. By this arrangement the air is constantly heated and circulated. Just inside the hole in the left wall of the baffle box is the element of the thermoregulator *T* used for automatically controlling the air temperature. Bolted to the left furnace wall, but electrically insulated from it, is the stop ring *R* through which the specimen assembly passes before extending through the baffled hole in the furnace wall to the outside. This ring is part of the electrical apparatus used as an automatic shut-off when the specimen breaks.

The specimen assembly which forms the cantilever beam consists of the test specimen *S*, shown in detail in Fig. 2, the steel extension *X*, and the connecting nut *D*. The reduced section of the specimen is turned with a tool swung on a 36-in. radius. The dimensions of the tool establish the $\frac{3}{8}$-in. fillets at either end of the reduced section. The inner end of the steel specimen extension is taper bored to receive the tapered end of the specimen which is drawn in tightly with the connecting nut. The outer end of the extension is turned to fit the bore of the self-aligning bearing of the variable eccentric *E*, which is keyed concentrically to the drive-motor shaft. With the variable eccentric set at a given deflection, the end of the specimen assembly is moved in the path of a circle when the drive-motor shaft is rotated. This motion of the specimen assembly will be referred to as oscillatory. The constant-deflection method of loading provides for one complete reversal of

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[3] H. F. Moore and N. J. Alleman, "Progress Report on Fatigue Tests of Low-Carbon Steel at Elevated Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part I, p. 114 (1931).

stress with each revolution of the motor shaft as indicated to the nearest 100 by the worm-driven counter *N*.

The variable eccentric *E* was designed to counter-balance automatically the centrifugal force of the oscillating specimen assembly on the eccentric bearing. By removing metal from the motor end of the bearing cavity, the eccentric was made dynamically off balance so that for any deflection the off-balance force of the eccentric would be equal and opposite to the radial force necessary to keep the end of the specimen assembly in its circular path. Without such a design the radial load on the motorshaft would produce excessive vibration.

Figure 3 is the wiring diagram for one of the units from which the electrical equipment will be explained. The power supply for the test units is 220-v., 60-cycle, three-phase alternating current. Control over each unit is attained by means of the start-stop switch which operates in conjunction with the magnetic switch. When the start button is pressed, the fan motor is started and power is available to the switches controlling the drive motor and the strip heaters. Heater *a* is always controlled by the thermoregulator through the temperature-control relay while *b* can also be automatically controlled if desired. Heaters *c* and *d* are either on or off all of the time, depending on the temperature to be maintained. The stop ring is connected through the magnet coil of the relay to the 12-v. secondary of the transformer, one end of which is grounded. When the specimen breaks, the extension touching the stop ring completes the circuit to ground, causing the magnetic switch to open, thus disconnecting the entire unit from the power supply.

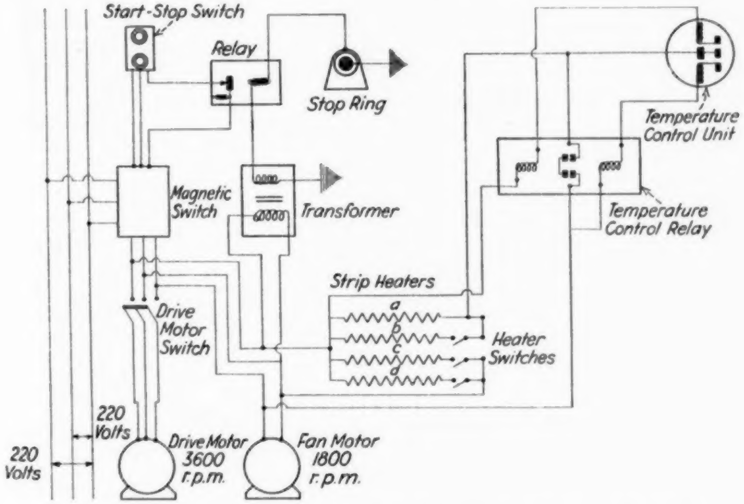

FIG. 3.—Wiring Diagram for High-Temperature Fatigue Machine.

TEMPERATURE MEASUREMENT AND CONTROL

For the results of elevated-temperature fatigue tests to be reliable, not only must the imposed stress be known accurately, but also the temperature of the test specimen must be maintained at the desired value. In order to determine how well the latter requirement can be carried out, consideration must be given to the means of temperature measurement, the temperature distribution and the limitations of the temperature-control apparatus.

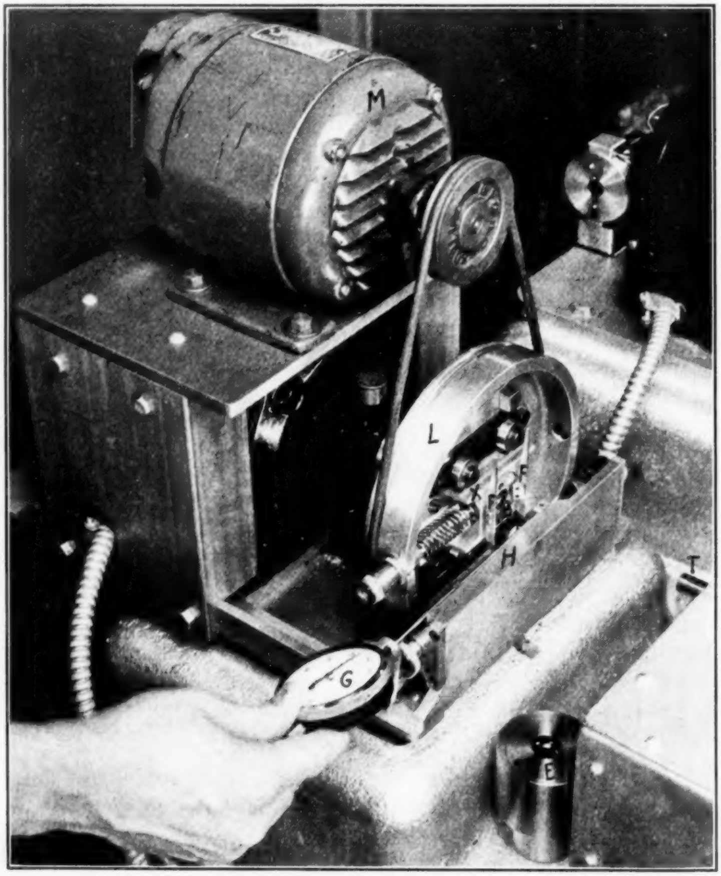

FIG. 4.—Calibration Apparatus for High-Temperature Fatigue Machines.

Numerous temperature surveys have been made using a standard specimen provided with four embedded thermocouples, one located in the shank of the specimen and the other three spaced along the reduced section. Air temperature was also measured at the element of the thermoregulator with a thermocouple. These surveys, which were made at specimen temperatures ranging from 300 to 500 F., using various strip heater arrangements, showed that by providing each test specimen with a thermocouple embedded in the cylindrical shank, the temperature of the specimen in the vicinity of the section of maximum stress can be controlled within ±2 deg. Fahr. The difference between the shank temperature and reduced section temperature is obtained from the temperature surveys.

The four 250-watt electric strip heaters have enough heating capacity to raise the specimen temperature to about 950 F., but the thermoregulator is capable of controlling only from room temperature to 600 F. Revision of the temperature-regulation apparatus would be necessary if test temperatures exceeding 600 F. were to be used.

CALIBRATION APPARATUS

The magnitude of the maximum stress imposed on the test specimen in terms of the deflection of the beam assembly cannot be calculated accurately by an analytical solution because of the nature of the beam and the difficulties encountered in determining the moduli of elasticity at the elevated temperatures. For this reason the calibration apparatus shown in Fig. 4 was developed. Its purpose is that of determining the load at the variable eccentric bearing corresponding to the resulting deflection of the beam. Use of this load and the flexure formula then can be made to determine the stress at any cross-section of the specimen.

In order to eliminate any creep effect in obtaining the load-deflection relationship, the calibration apparatus was designed so that the specimen might be loaded and the resulting deflection measured while the machine is in operation. In Fig. 4 the calibration apparatus has been shifted along the base keyway away from the working position to show the details of the load wheel *L*, the feeler gage assembly *H*, and the auxiliary drive motor *M*. For calibration tests the entire assembly is slid into position so that the end of the beam *T*, protruding from the furnace wall, enters the bore of the self-aligning bearing *B* carried by the load-wheel crosshead *X*. The load-wheel spring *S*, which bears against the crosshead, can be adjusted to the desired loads as indicated by the deformation of the spring measured with a dial gage, the stem of which is placed inside the spring assembly. Rotation of the load wheel then applies a radial load to the end of the specimen assembly. Although the load wheel is mounted on the drive-motor shaft in place of the variable eccentric *E*, it is revolved at about 300 r.p.m. by a belt drive from the auxiliary motor rather than at 3600 r.p.m. by the drive motor.

The resulting amplitude of oscillation, which is twice the deflection, is measured with the feeler gage assembly *H* in front of the load wheel. The two electrically insulated vertical feelers *F* are mounted on individual horizontal slides which are actuated through lead screws by the knurled knobs *K*. Contact between the feeler points and the oscillating specimen assembly is indicated by completion of a circuit through $\frac{1}{4}$-watt neon lamps in the 110-v. a.c. line. Amplitude of oscillation is measured by the dial gage *G*, supported on one slide. The tip of the gage rests against a rod carried by the other slide.

CALIBRATION AND TEST PROCEDURE

The procedure in running the fatigue tests is that of using one of the group of specimens of the material to be tested to determine the nominal stress-amplitude of oscillation relationship at the test temperature. This relationship is then made use of to impose the desired stresses on the remaining specimens to be tested in fatigue. Nominal stress is calculated as the maximum fiber stress at the section of minimum diameter. The relationship between nominal stress and load-spring deformation is easily found by combining the flexure formula and the load-deformation calibration of the load spring.

The specimen to be calibrated is heated to the test temperature in the machine which has been equipped with the calibration apparatus. Increments

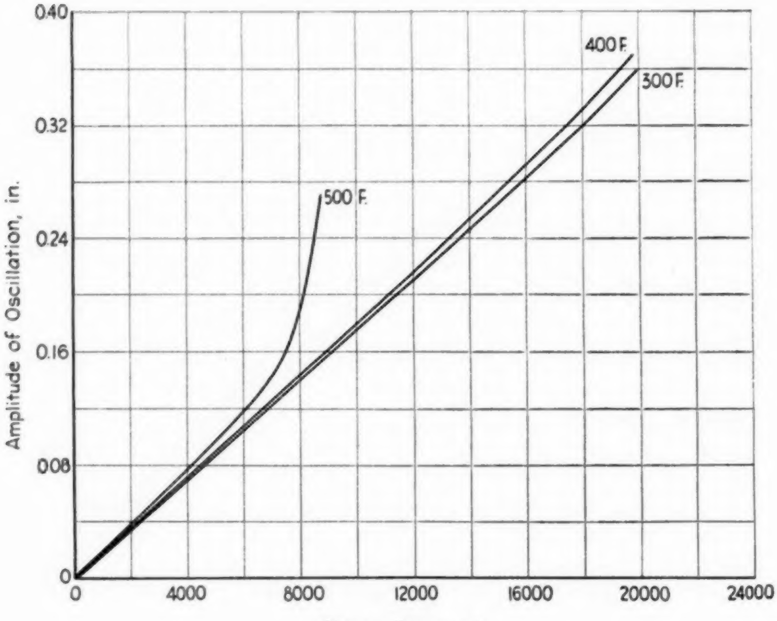

FIG. 5.—Stress-Amplitude of Oscillation Curves for High-Temperature Fatigue Tests on Alcoa 17S-T Wrought Aluminum Alloy.

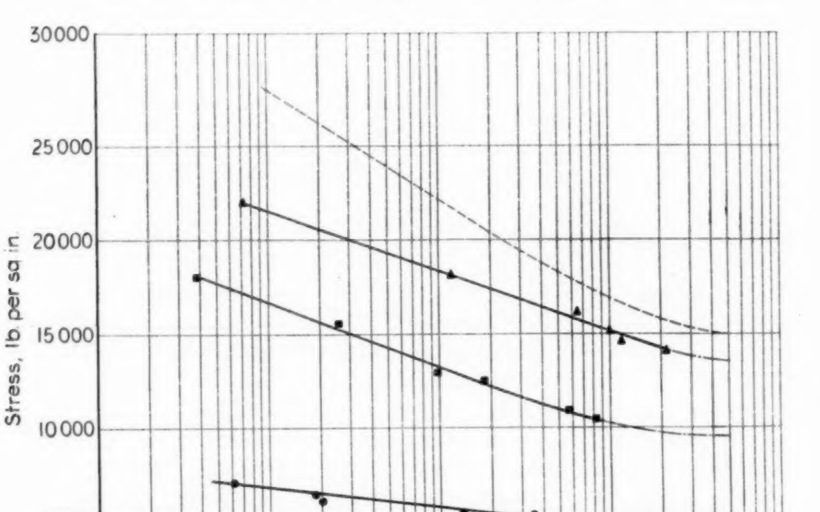

FIG. 6.—Elevated and Room Temperature Fatigue Tests for Alcoa 17S-T Wrought Aluminum Alloy.

of stress are applied by adjusting the load spring, the deformation of which is measured only in the horizontal plane to eliminate the effect of the dead weight of the specimen assembly and load-wheel crosshead. After each spring adjustment, the load wheel is rotated by the auxiliary motor and the resulting amplitude of oscillation is measured by running up the feelers until they contact the oscillating specimen extension and then backing them off until contact no longer is made. Plotting the data thus obtained results in the nominal stress-amplitude of oscillation relationship.

Having calibrated one specimen of the lot to be tested, determination of the *S-N* curve is the next objective. The calibration apparatus is replaced by the variable eccentric so that fatigue tests to fracture may be conducted at 3600 cycles of stress per minute. The variable eccentric is set to impose the desired stress by the use of a flat-tipped dial gage which measures the amplitude of oscillation at the same position at which it was measured with the feeler gage assembly during calibration. Each test is started from a zero counter reading and is automatically concluded when the specimen breaks.

TABLE I.—PROPERTIES OF 17 S–T ROD AT ROOM AND ELEVATED TEMPERATURES.[a]

	75 F.	300 F.	400 F.	500 F.
Heating period before tension test, days	....	250	200	200
Tensile strength, lb. per sq. in.	58 965	40 200	25 000	12 750
Yield strength (0.2 per cent set), lb. per sq. in.	36 000	33 500	20 000	9 700
Elongation in 2 in., per cent	24	17	25	38
Heating period before fatigue test, days	....	200	100	100
Endurance limit, lb. per sq. in.[b]	15 000[c]	13 500	9 500	4 500
Percentage of room-temperature endurance limit	100	90	63	30
Endurance ratio[d]	0.25	0.34	0.38	0.35

[a] Tensile properties taken from "Properties of Wrought Aluminum Alloys at Elevated Temperatures," by F. M. Howell and D. A. Paul, *Metals and Alloys*, October, 1935, p. 284.

[b] Endurance limit based on 500,000,000 cycles.

[c] Tested in R. R. Moore rotating-beam fatigue machines.

[d] Ratio of endurance limit to tensile strength.

CALIBRATION AND FATIGUE TEST RESULTS

The type of stress-amplitude curves obtained with the calibration apparatus for these high-temperature fatigue machines is shown in Fig. 5. These particular curves are the results of calibrations made on a wrought alloy of the duralumin type (Alcoa 17S-T) at 300, 400 and 500 F., but are representative of the general type of curves obtained on other aluminum alloys. The change in slope of the curves with increase in temperature is indicative of the decrease in modulus of elasticity.

Fatigue tests are run at amplitudes of oscillation chosen from Fig. 5 to give the desired stress in the test specimen. This stress and the counter reading at specimen failure are plotted to semi-logarithmic coordinates as shown for Alcoa 17S-T in Fig. 6. These fatigue curves are accompanied by the room-temperature, rotating-beam fatigue curve, obtained using machines of the R. R. Moore type, to show more completely the effect of the elevated temperatures on the fatigue properties.

The relationship between the fatigue properties and the tensile properties are shown in Table I. As indicated in this table, the fatigue specimens were conditioned for the fatigue tests by heating for a long time at the testing temperature. This stabilizing treatment is necessary to insure no appreciable change in physical properties due to the prolonged heating during the fatigue tests. The heating period necessary to reach approximate physical property equilibrium was determined from tension tests made after prolonged heating.[4] The values given for the elevated-temperature endurance limits are based on the incomplete fatigue curves projected to 500,000,000 cycles. It is of considerable interest to note the apparant sharp increase in endurance ratio from 0.25 at room temperature to an average value of 0.36 at the elevated temperatures. In calculating the room-temperature endurance ratio, no allowance has been made for the fact that the temperature of the R. R. Moore rotating-beam fatigue specimen is somewhat greater than room temperature because of the heat conducted from the bearings and the heat generated in the specimen. Use of the tensile strength of the material at the temperature of the fatigue test specimen would probably raise the endurance ratio slightly. It is believed that too close comparisons cannot be made between the room-temperature and elevated-temperature endurance ratios because the fatigue tests were conducted in different type machines. Room-temperature tests using the elevated-temperature machines are planned but have not actually been made at this time.

ACCURACY OF CALIBRATION AND TEST DATA

The test results obtained with this equipment are accurate and consistent. An effort has been made to investigate all possible sources of error in calibration and test. One of the greatest of these is in the alignment of the drive motor. In order to insure complete stress reversal the amplitude of oscillation must be twice the maximum deflection of the specimen assembly. This is accomplished by substituting a straight piece of steel drill rod for the specimen assembly and aligning the drive motor horizontally, vertically and angularly so that its axis and that of the drill rod form a straight line. The center of oscillation then corresponds to the zero stress position of the specimen assembly. The maximum vertical deflection of the aligning rod under its own weight is less than 0.001 in.

The method of stress calculation also provides sources of error. An investigation of the stress distribution in the specimen showed that the maximum fiber stress is about 2 per cent greater than the nominal stress (maximum fiber stress at section of minimum diameter). The nominal stress, however, is calculated from room-temperature dimensions instead of the dimensions of the heated specimen which should be used. The error thus encountered depends upon the thermal expansion of the specimen. For aluminum alloys at 500 F., this expansion reduces the stress about 2 per cent so that at this particular temperature these errors cancel each other.

Specimen size cannot be held to exactly the specified dimensions. A maximum variation of ±0.002 in. has been found in the 150 specimens machined until the time of this report. Calculations show that use of the calibration curve for a specimen of specified dimensions in determining the stress in other specimens within the machining tolerances introduces an error not exceeding 0.5 per cent.

Two investigations have been carried out to determine the effect of the centrifugal force of the mass of the oscillating specimen assembly on the stresses in the specimen. One proved that the amplitude of oscillation for a given eccentric setting remained the same whether the eccentric was turned over slowly by hand or driven at a speed of 3600 r.p.m. The other showed that the shear stress

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[4] F. M. Howell and D. A. Paul, "Properties of Wrought Aluminum Alloys at Elevated Temperatures," *Metals and Alloys*, October, 1935, p. 284.

due to the centrifugal force increases the fiber stress about 1 per cent.

The errors arising from personal elements connected with running the tests, such as aligning the motors, setting the eccentrics and obtaining values from the calibration curve, should not exceed 2 per cent. Considerations of these errors along with the other experimental errors, some of which are counteractive, indicate that the total error in any test should not exceed 3 per cent.

Acknowledgment.—Acknowledgment is due to Mr. R. L. Templin, Chief Engineer of Tests, Aluminum Company of America, who was primarily responsible for the development of these machines and under whose direction the test work is being carried out.

DISCUSSION

Mr. H. F. Moore[1] (*presented in written form*).—The fatigue testing machine described by Messrs. Howell and Howarth is an elegant and, as judged by its calibration, accurate machine of the constant-strain (or constant-deflection) type. It differs in type from the machine developed at the University of Illinois, which utilizes a rotating spring to apply load to the specimen, and, by checking up the deflection of this loading spring at intervals, a fatigue test can be run under very nearly constant-stress conditions. Whether there is any difference between fatigue limit determined by a constant-strain machine and one determined by a constant-stress machine is not certain, although a few tests at the University of Illinois indicate that there may be a slight difference. The results of tests of Alcoa 17S-T run on the machine described by Howell and Howarth at room temperature would be of interest for comparison with the results of the tests of that metal in an R. R. Moore rotating-beam fatigue testing machine.

Mr. H. J. Gough.[2]—In offering my congratulations to the authors for an excellent paper, I would express a need for caution in interpreting the results of fatigue tests made at elevated temperatures, especially when reversed flexural stressing is employed. The data presented, when plotted in the form of *S-N* curves, show no real approach to a limiting range of stress, although the dotted extensions to the curves of Fig. 6 suggest that the authors expect these curves to become parallel to the *S* axis. Such expectation may seem warranted from experience with the majority of metals tested under fatigue stressing at air temperature, although it will be recalled that lead forms a notable exception, no fatigue limit having yet been found for that metal. As the test temperature is raised, an increasing number of metals and alloys tend to behave in a manner similar to lead at air temperature; in fact, the term "elevated temperature" has no general meaning and probably is a quantity which must be considered, in any particular case, in relation to the melting point of the metal and the absolute zero of temperature. Our experience has taught us that when fatigue tests are made at a temperature within the "creep field" of the material investigated, an erroneous conclusion may easily be drawn if specimen endurances only are considered. There is no doubt that a "speed effect" exists when creep is in operation, tending to suppress and delay total deformation and fracture. For instance, tests at the National Physical Laboratory have clearly shown that steels and light alloys may be subjected without fracture to many millions of cycles of a range of *pulsating direct* stresses, the *minimum* stress of which cycle, if applied as a static load, would produce extension and fracture in a comparatively short time. In such cases, it is our practice to record the changes in total length of the specimen during the fatigue test;

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[1] Research Professor of Engineering Materials, University of Illinois, Urbana, Ill.

[2] Superintendent, Engineering Dept., National Physical Laboratory, Teddington, Middlesex, England.

if creep is still in operation, especially if it is of such an amount as to indicate that static failure is inevitable under prolonged testing, the fact that the specimen is still unbroken in the fatigue machine can have little real significance in design where the fatigue stressing may be intermittent or of lower frequency. There is an increased probability of anomalous results if such high-temperature fatigue tests are carried out using flexural stressing.

I, therefore, wish to urge that, in the field of testing at elevated temperatures, the creep and fatigue properties must be studied jointly and correlated, if reliable data are to be obtained.

MESSRS. F. M. HOWELL[3] and E. S. HOWARTH[3] (*authors' closure, by letter*).—Inasmuch as the testing machines with which this paper is concerned have been operating only a short time, extensive data have not been obtained. Although tests at room temperature have not yet been carried out in these elevated-temperature fatigue testing machines, plans have been made to conduct such tests so that a comparison can be made with the results of tests conducted in R. R. Moore fatigue testing machines. Such a comparison may not show clearly the relationship between endurance properties as determined by constant-deflection and constant-stress methods because of the fact that heat generated in the bearings of the R. R. Moore fatigue machines raises the temperature of the test specimen considerably above room temperature while in the machine which this paper discusses, such is not the case. It might be worth while to repeat the specimen calibration at various times during the progress of the test to determine whether or not there are any changes in the load-deflection relationship.

The authors agree whole-heartedly with Mr. Gough in his discussion of the application of the results of fatigue tests. Quite naturally in the design of any mechanism or structure, all of the properties of the material must be considered. In elevated-temperature applications, consideration is given primarily to the creep and fatigue characteristics of the material under the loading conditions assumed.

As stated in the paper, the fatigue curves are given merely for illustrative purposes. Possibly the dotted extensions of the curves are not good estimates of what will be obtained. It is planned to carry the tests out to larger numbers of cycles but, of course, there is no assurance that the curves will become horizontal.

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[3] Engineer of Tests, and Research Engineer, respectively, Aluminum Research Laboratories, Aluminum Company of America, New Kensington, Pa.

THE RELAXATION OF COPPER AT NORMAL AND AT ELEVATED TEMPERATURES[1]

By John Boyd[2]

Synopsis

As increasing amounts of information on the subject of creep are being accumulated, relaxation, which is a manifestation of creep, is beginning to attract more and more attention. The phenomenon of relaxation is exhibited when a material is stretched to, and then held at, a given extension. Creep immediately begins to take place, and this tendency to elongate causes the stress within the material to decrease. This reduction in stress may be observed particularly in the case of bolts. It is common knowledge that bolts, which originally have been drawn up tightly, frequently are found to have loosened considerably after long service at high temperatures. A knowledge of the laws which govern the decrease of stress should be of much practical interest, but in addition it should also bring about a clarification and an extension of the whole picture of creep. The present paper is intended to present an approach to the problem of relaxation based on general principles. To obtain the experimental data a special automatic machine was designed and built. Copper was employed in testing because, being a fairly pure material, its use seemed to present a means of studying the problem without giving rise to involved additional effects. The paper furnishes experimental information upon the interdependence of such factors as stress, temperature, time, and the amount of cold work.

Introduction

With the present-day tendency in various industries to employ higher temperatures, it is becoming of greater importance to investigate the behavior of metals at elevated temperatures more extensively than was necessary in the past.

In the study of the mechanical properties of metals at elevated temperatures, the method most usually adopted is the long-time creep test in which the test specimen, held at constant temperature, is allowed to creep (or elongate) under the action of a constant load.

When a metal is subjected in practice to conditions similar to those under which it is tested in the ordinary long-time creep test, such tests furnish good and reliable information. In many applications, however, the conditions of service are somewhat different from those which prevail in the usual creep test. For example, bolts tightened on two relatively rigid flanges and held at a given temperature will have their length kept practically constant but their stress will be found to decrease. The question then arises as to whether the

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[1] The work reported in this paper was done under a grant received from the Engineering Foundation during the years 1935–1936 and was carried out with the facilities offered by the Research Laboratories of the Westinghouse Electric and Manufacturing Co. in cooperation with the University of Pittsburgh. The problem was suggested by Dr. A. Nadai of the Westinghouse Research Laboratories who influenced the work as it progressed.

[2] Engineering Foundation, New York City. Present connections: Research Engineer, Research Laboratories, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

decrease in stress can be predicted from creep tests. To answer this query it is of course necessary to determine the manner in which the stress decreases by conducting tests under the same conditions as those to which a bolt is subjected. Such an investigation may also provide an answer to the converse question as to whether, from a knowledge of the decrease of stress at constant length, the creep under constant stress can be determined.

The phenomenon of the decrease of stress at constant length has been called relaxation. Some investigation of this phenomenon has been made but few of the methods used have attempted to produce pure relaxation conditions. In the majority of instances, the goal of the experimentation has not been to describe the laws of relaxation but to obtain creep data in a more rapid way than was possible from tedious long-time creep tests.

Such was the aim in the experiments of Rohn,[3] where an apparatus kept the length of a loaded test specimen constant by lowering the temperature so that the thermal contraction due to cooling just compensated for the extension due to creep. This was probably one of the first experimental approaches to the general problem, but the method gives results that are difficult to interpret.

Barr and Bardgett[4] introduced a different scheme in which the test specimen and a calibrated weigh-bar were stretched together in a rather rigid frame. As the test specimen crept, the weigh-bar would contract elastically and the stress would decrease. Disregarding any flexibility in the frame, this method consists essentially in keeping the total length of the test specimen and weigh-bar constant. These are not pure relaxation conditions for, even with a perfectly rigid frame, the test specimen must become longer if the stress in the weigh-bar is to decrease. In addition there are the disturbing effects due to any flexibility of the frame and changes of length caused by temperature variations.

More recently, direct measurements on relaxation in bolts under tension at high temperatures have been made by N. L. Mochel and C. R. Soderberg at the South Philadelphia Works of the Westinghouse Co. This work is still in progress.

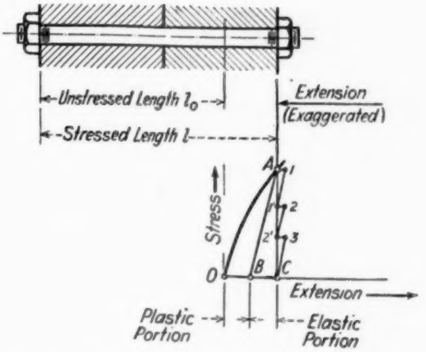

FIG. 1.—Stress Extension Diagram for a Bolt Tightened on Rigid Flanges.

Relaxation tests have also been made, recently, at Harvard University with an apparatus operating in torsion, which was designed by Doctor Nadai and Mr. R. Beeuwkes. Torsion measurements were suggested in order to eliminate disturbances due to thermal expansion of the frame. Results from this work have not as yet come to our attention.

During the past two years, relaxation tests have been made at East Pittsburgh with an apparatus designed to eliminate the principal difficulties of former methods. Special attention has been directed toward fulfilling the conditions of pure relaxation, that is, measuring the de-

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[3] W. Rohn "Creep Stress Determinations," *The Metallurgist*, February, 1930, p. 22.

[4] W. Barr and W. E. Bardgett "An Accelerated Test for Determining the Limiting Creep Stress of Metals," *Proceedings*, Inst. Mechanical Engrs. (British), Vol. 122, p. 285 (1932).

crease in stress while the length of the test specimen is carefully kept constant. The principle of this special apparatus may be best understood by referring to Fig. 1. Let a bolt of a given material be stretched by tightening, from the unstressed length l_o to the stressed length l. The stress-extension curve is then shown as OA. If the load were then removed, the approximately straight line AB would appear as the unloading curve. The elastic portion of the extension, BC, would be regained, but the plastic part, OB, would remain as a permanent deformation. However, suppose the length l of the bolt were held constant for an indefinite time; the stress would then decrease but the extension would stay constant so that the unloading line would be represented by the vertical line AC. In the experiments to be described, the line AC, which represents the conditions of relaxation, is approximated by a series of very small steps (those shown in the figure are very much exaggerated). The principle by which this was carried out in the special apparatus is as follows. The test specimen is first stretched to the length l after which the applied load causes the test specimen to creep, so that, at first, extension takes place at constant stress (line A–1 in Fig. 1). A portion of the load is then removed and the specimen contracts elastically along line 1–1′. The mechanism is designed to bring the length back to its initial value l. Since creep also takes place at this reduced stress, extension again continues along line 1′–2, but at a slower rate. At point 2, more of the load is removed and the cycle repeated indefinitely. By making the steps very small a true relaxation test is approximated and the decrease in stress with time may be observed.

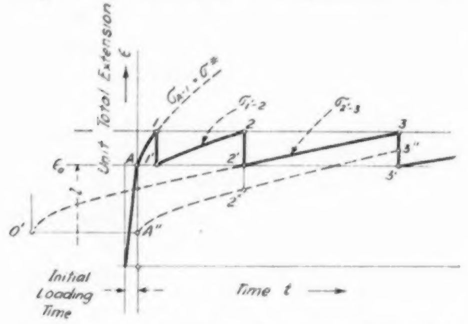

FIG. 2.—Constant Extension Approximated by an Interrupted Creep Test.

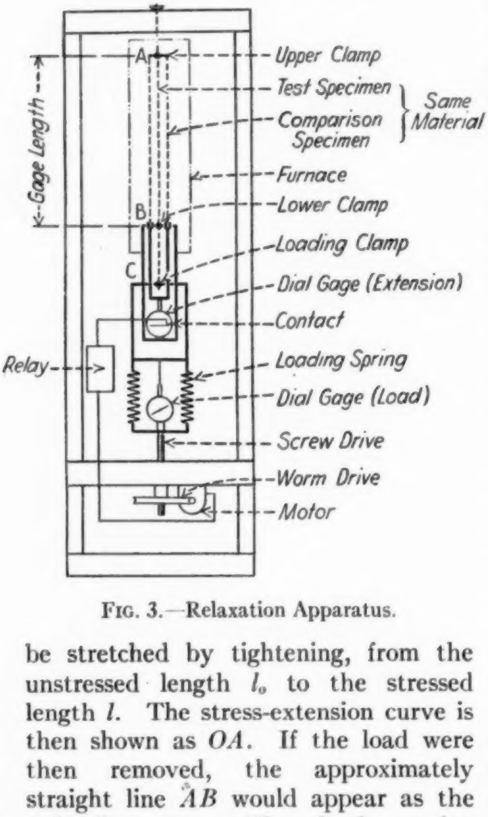

FIG. 3.—Relaxation Apparatus.

Figure 2 illustrates the relation between extension and time for a succession of finite unloadings such as were considered in Fig. 1. The lines A–1, 1′–2, 2′–3, etc., are the creep curves corresponding to the lines having similar marking in the latter figure.

APPARATUS

Figure 3 is a diagrammatic sketch of the apparatus used. The gage length (A to B) is 20 in. and the specimens are $\frac{3}{16}$ in. in diameter. Comparison specimens are of the same material and diameter as the test specimen and equal in length to distance *AB*. Extending from point *B* downward, four steel rods carry the relative motion of points *A* and *B* out of the furnace to the dial gage. The gage is equipped with a contact which engages the pointer at a predetermined value of the extension. Establishing contact cuts out a 4 or 5-v. grid bias on a grid glow relay which in turn is connected to start the motor. Load is applied to the specimen at point *C* through the small frame and the springs. It is varied by the motor which drives the screw up and down, thus extending or releasing the springs.

In normal operation the apparatus is quite sensitive. The dial gage will respond and cause the motor to operate on a change of length of about 1/75,000 of an inch over the gage length of 20 in. (This corresponds to about 10 lb. per sq. in. change in stress.) The length of the test specimen is thus kept constant to within this limit.

Considerable care was exercised in the design and construction of the furnace to insure uniform heat distribution and constant temperature. The furnace is 40 in. long, thus providing 10 in. of heated length beyond each end of the 20-in. gage length of the test specimen. The heating element consists of 15 units, each with external taps for temperature regulation. Two concentric monel metal tubes, separated by an air space, surround the test and comparison specimens. Temperature may be kept constant to ±0.5 deg. Cent. with regard to time and to ±1 deg. Cent. along the gage length.

In all tests, the specimen, with extensometer in place, was put in the heated furnace and allowed to remain unloaded for 1 hr. before the testing load was applied. About 50 min. were required to reach a constant temperature. Temperature was controlled by a device employing the relative difference in expansion between a quartz rod and a nickel tube. This operated a vacuum switch connected so as to interrupt the entire furnace current (4 to 8 amp.). The operation was very satisfactory and was practically independent of changes in room temperature.[5]

PREPARATION OF TEST MATERIAL

Copper was chosen as a test material because of its importance in electrical machinery as well as its relative simplicity. The material used in a large number of the room temperature tests was taken from the same reel of hard-drawn 0.204-in. diameter copper wire. This was cut to length, heated by passing a current through it so that it could be readily straightened, and then annealed by heating to 570 C. for 2 hr. in an electric furnace with a hydrogen atmosphere.

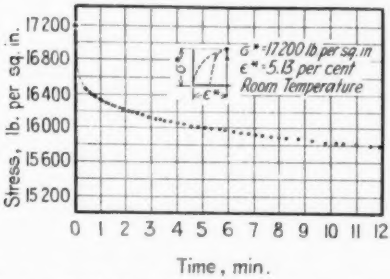

FIG. 4.—Typical Relaxation Curve for Copper.

From the results of tension and other tests, it later appeared that this copper was not entirely oxygen-free and that it suffered a slight amount of embrittlement on the surface, for its strength was found somewhat reduced and its creep rate was observed to be increased. When this was discovered, a new copper was prepared but it was felt that the large amount of information obtained from the first copper was quite useful and should be included in the report.

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[5] Recommendations as to the design of the temperature control were made by J. R. Gier of the Research Laboratories.

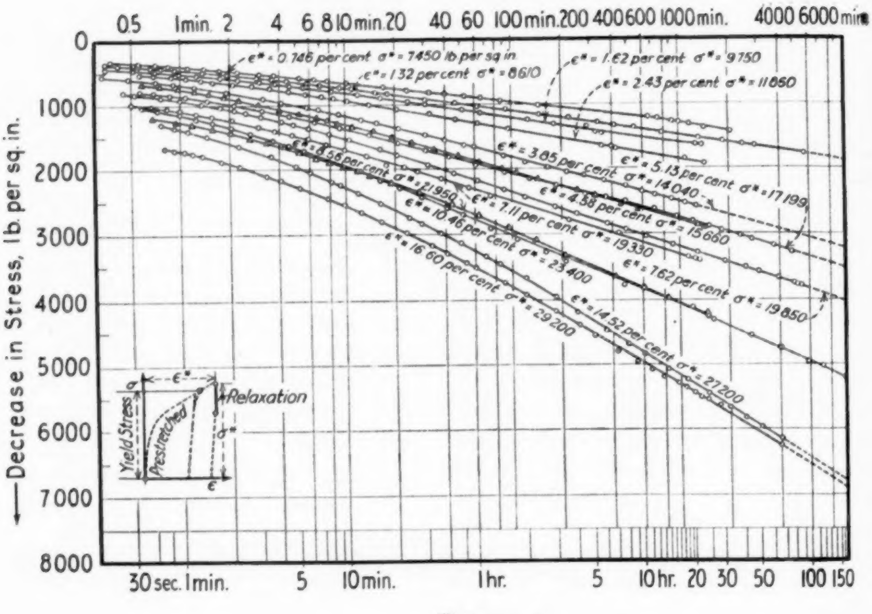

FIG. 5.—Relaxation of Copper at Room Temperature
Tested above the yield point. First lot of test material.

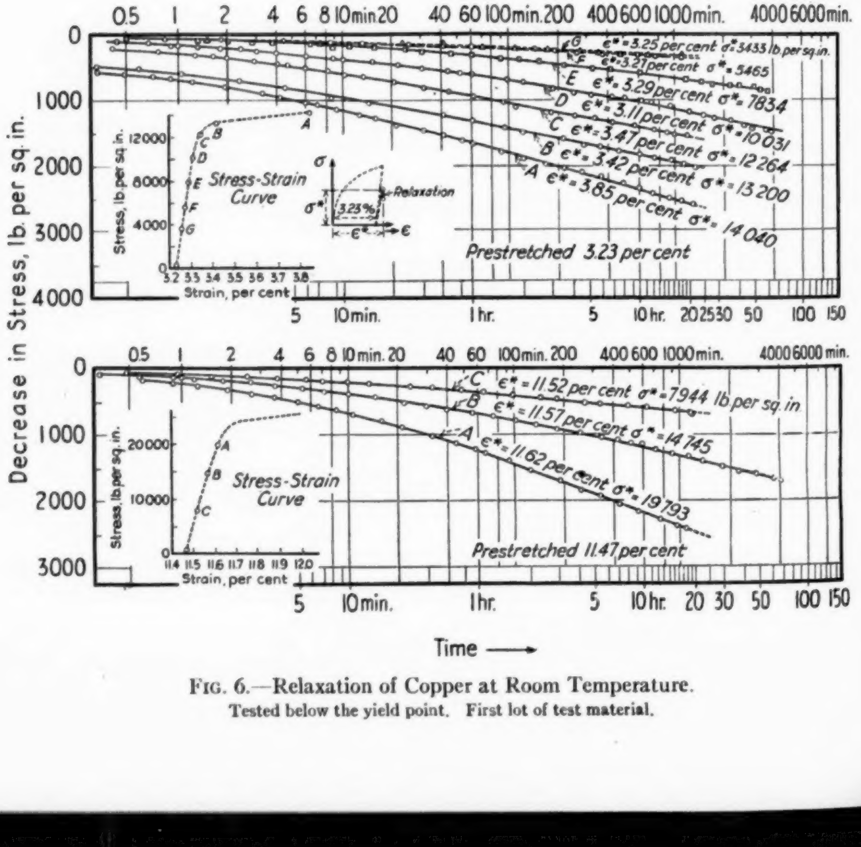

FIG. 6.—Relaxation of Copper at Room Temperature.
Tested below the yield point. First lot of test material.

The curves shown in Figs. 4, 5, 6, 7, 8, and 9 are based on this material.

For all other tests, the second lot of copper was used. This was carefully annealed between different passes in the drawing operation and then annealed in nitrogen at 650 C. for $\frac{3}{4}$ hr. before it was prestretched preparatory to testing. A small number of tests was made with this second copper to check the results obtained with the first. These were in substantial agreement with the previous tests and indicated that only the tensile strength of the first material was somewhat reduced due to the embrittlement on the surface which had reduced the area effective in carrying stress (compare Figs. 7 and 15).

TEST RESULTS

Figure 4 shows a typical relaxation curve as it appears when plotted on ordinary stress-time coordinates. It will be observed that during the first portion of the test, creep is so rapid that the machine had to unload continually for about 20 sec. in order to bring the length of the test specimen back to its "constant length" value. The plotted points represent readings obtained each time the machine performed an unloading. Although this is the ordinary form of expressing the results, inferences drawn from certain theoretical considerations, as well as the need of compressing the time scale, led to plotting almost all of the data semi-logarithmically.

To secure copper having various amounts of initial strain it was necessary to prestretch the test specimens in another machine, so that the strain registering capacity of the relaxation machine would not be exceeded.

With a material in a prestretched or cold-worked condition, the application of loads below the yield stress (see small sketch Fig. 5) is accompanied by a certain amount of relaxation or creep, but if the load exceeds the yield value, very much larger effects are observed. To facilitate the description of tests conducted under these different conditions, the two following expressions have been introduced here. When the prestretched copper was loaded to a stress below its yield stress it is described as being "tested below the 'yield stress'" or point. When it was loaded to a stress above the yield value it is described as being "tested above the yield stress."

Figure 5 shows a number of relaxation curves plotted semilogarithmically. All refer to copper tested above the yield stress. The values of the various initial stresses and strains are marked on each curve. It is seen that the curves gradually straighten out as time increases.

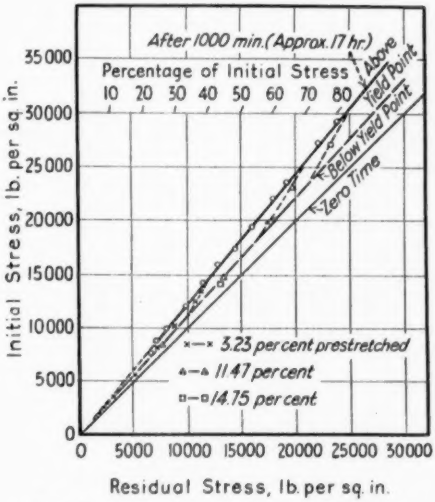

FIG. 7.—Relaxation of Copper at Room Temperature.
First test material.

Figure 6 shows several relaxation curves for copper prestretched in the amounts of 3 and 11 per cent, approximately. Again the curves straighten out with increasing time.

Figure 7 has been plotted from the results of Figs. 5 and 6. The points plotted with a circle are taken from

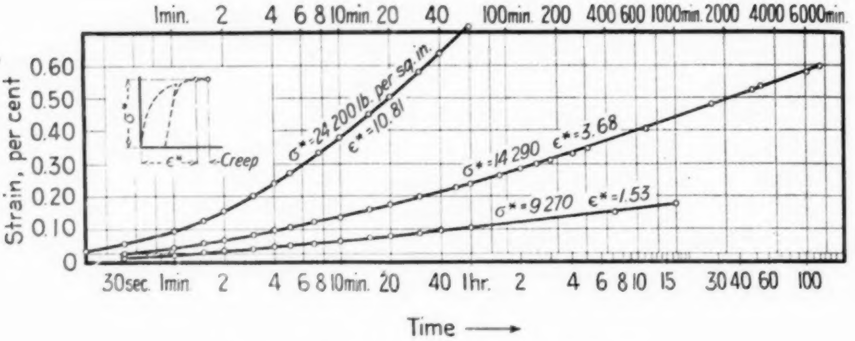

FIG. 8.—Creep of Copper at Room Temperature.
Tested above the yield point. First lot of test material.

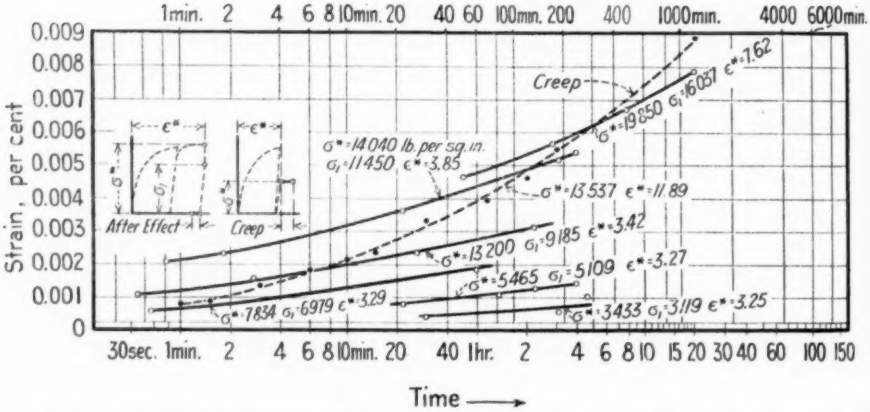

FIG. 9.—Aftereffect in Copper and Creep Below the Yield Point at Room Temperature.
First lot of test material.

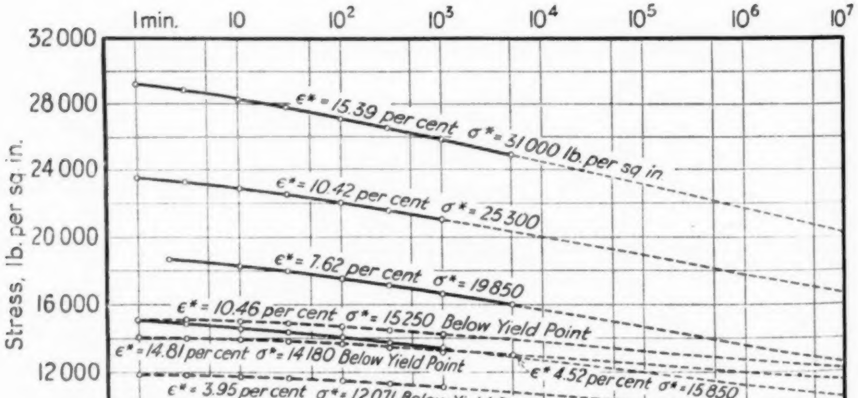

FIG. 10.—Relaxation of Copper at Normal Temperature.
Tested above and below the yield point.

Fig. 5 and represent the residual, remanent, or remaining, stress at a given time (1000 min., or about 17 hr.) for copper tested above the yield stress with the given initial stress. A rather unexpected result is that these points lie very nicely on a straight line. The other points are taken from Fig. 6. Here it will be seen that as long as the material is initially stressed to a load below the yield value, the amount of cold working is not very important. For example, suppose three specimens had been prestretched to 3, 11, and 14 per cent, respectively, and that each had been subsequently loaded to an initial stress of say 8000 lb. per sq. in., then the remanent stress after a given time would be substantially the same in each case. If the 3 per cent specimen had been brought to an initial stress of say 16,000 lb. per sq. in., the yield stress would

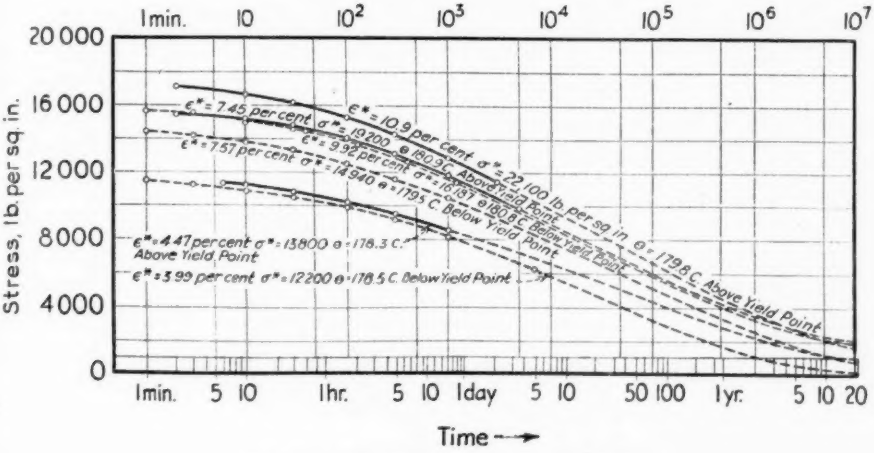

FIG. 11.—Relaxation of Copper at 180 C.
Tested above and below the yield point.

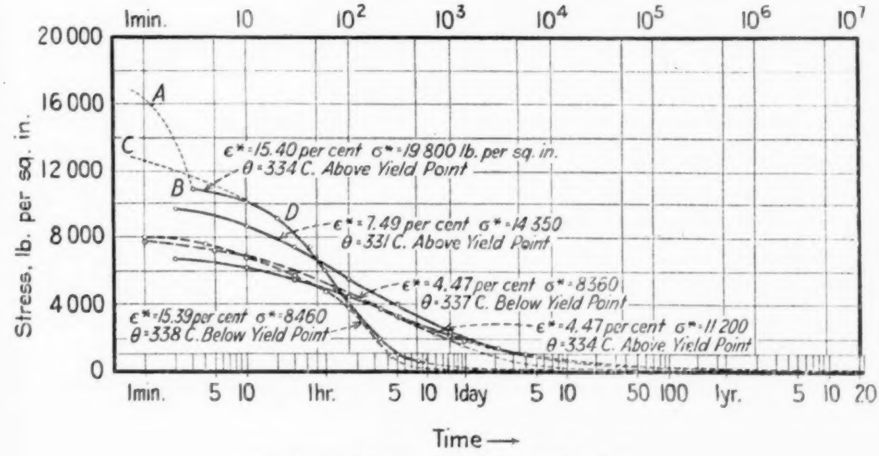

FIG. 12.—Relaxation of Copper at 330 C.
Tested above and below the yield point.

have been exceeded and it would have behaved as a bar tested above the yield stress. The curves shown in Fig. 7 are for 1000 min. but a similar group exists for any other amount of elapsed time.

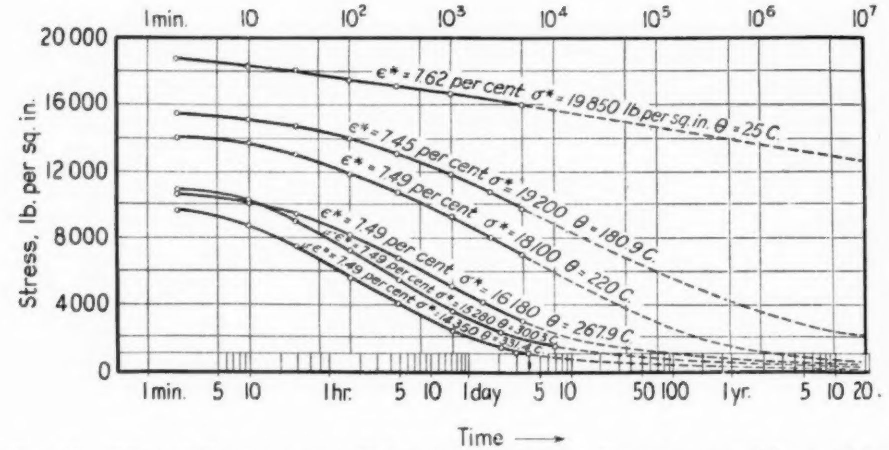

FIG. 13.—Effect of Temperature on Relaxation Copper for a Given Amount of Prestretching. Tested above the yield point.

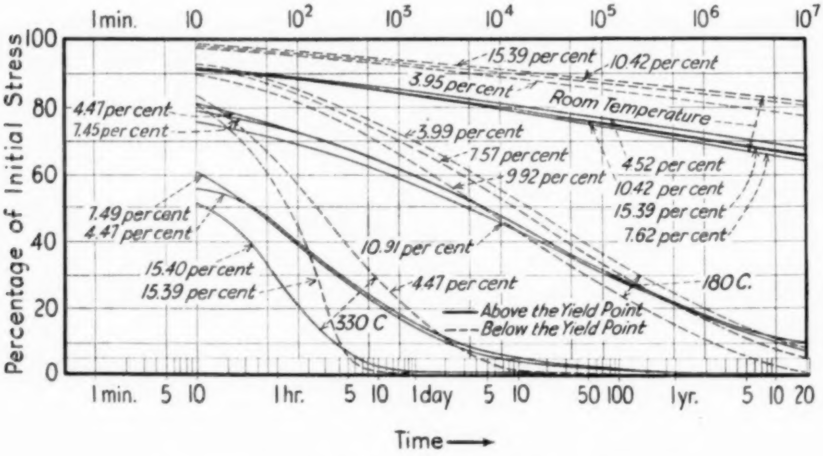

FIG. 14.—Relaxation of Copper at Various Temperatures.

To establish a correlation between relaxation and creep it was thought advisable to run creep tests on the same material in the same machine. The latter condition was made possible by removing the contact from the dial gage measuring the strain and putting it on the gage indicating the stress in such a way as to restore the initial load as the bar crept.

Figure 8 illustrates creep curves determined in the above manner for copper tested above the yield stress. The dash line in Fig. 9 is also a creep curve, but for copper tested below the yield stress. Figure 9 shows in addition the aftereffect or recovery which occurs when the load is removed. These latter curves were obtained after unloading at the end of various relaxation tests. The comparative magnitudes of the aftereffect and the creep can be seen in this figure.

After this series of comparative relaxation and creep curves had been obtained, the succeeding tests were all conducted with the second lot of copper prepared as previously described.

Figure 10 is a repetition of the room temperature relaxation tests using the new copper and plotting remanent stress against time. Most of the tests had a duration of from one day to a week. Extrapolation has been carried out to 20 yr. Figure 11 is similar to Fig. 10, but refers to a temperature of 180 C. Figure 12 shows relaxation at 330 C., with the rapid softening of the more highly prestretched material to be noted.

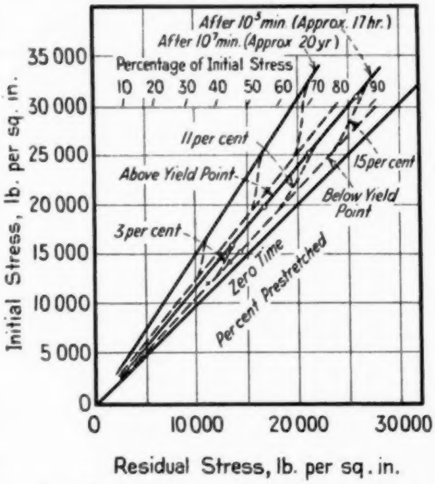

FIG. 15.—Relaxation of Copper at Room Temperature.

The manner in which temperature alters the relaxation of copper having the same amount of cold work is shown in Fig. 13. For the temperatures of 267, 300, and 330 C., an inflection point is clearly visible. The shape of these curves forms the basis for extrapolating the curves for 220, 180 C., and room temperature both in this figure as well as in Figs. 10, 11 and 12.

If the remanent stress is expressed in percentage of the initial stress and plotted against time it will be found that the curves for copper tested above the yield stress at room temperature with various amounts of initial stress all lie on practically the same line (see Fig. 14). The same is true for copper tested below the yield stress. Thus two lines in this plot may be used to represent the relaxation of copper at temperatures that are close to normal. For copper tested above the yield stress at room temperature an expression that fits those results quite well may be written in the form:

$$\sigma = \sigma^* [1 - 0.045 \log_{10} (1 + 2.93t)] \ldots (1)$$

where σ = the remanent stress in pounds per square inch,

σ^* = the initial stress in pounds per square inch, and

t = the elapsed time.

At 180 C. the lines for tests conducted above and below the yield stress are

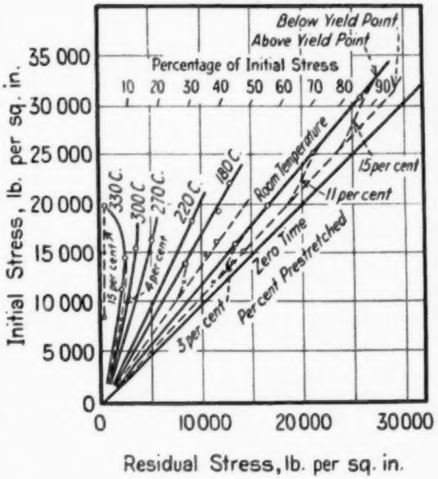

FIG. 16.—Relaxation of Copper at Various Temperatures.
After 1000 min. (approximately 17 hr.).

farther apart for the smaller values of elapsed time and both sets tend to straighten out, then reverse their direction of curvature as time increases.

When the temperature is raised to 330 C., the curves for copper tested above the yield stress and having different amounts of cold work no longer lie along one line but become considerably separated. The same is true in the case of copper tested below the yield stress. The cause of separation lies in the fact that the more highly cold-worked material softens more readily than that which is less cold worked.

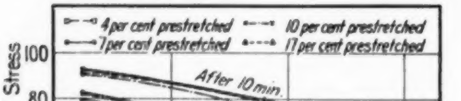

FIG. 17.—The Effect of Temperature on the Relaxation of Copper for Various Amounts of Prestretching.

In Fig. 15, the relation between remanent stress and initial stress is shown for room temperature. The curves for 20 yr. (extrapolated from Fig. 10) are of the same form as those for the observed time of 17 hr. If these results are compared with those of the first copper, very little difference will be noted (compare Figs. 7 and 15).

Figure 16 shows the remanent stress plotted against initial stress for various temperatures. With increasing temperature the lines can be seen to become more and more curved. There is also the disturbing effect produced by the more rapid softening of the material which has had the greater amounts of cold work.

Figure 17 shows the influence of temperature upon the relaxation of copper having different amounts of cold work. In this plot the remanent stress is shown as a percentage of the initial stress for different periods of elapsed time. From a practical point of view, the amount of relaxation taking place in 10 min. or even in 100,000 min. (approximately 70 days) would not be of as much interest ordinarily as that which would occur in 10,000,000 min. (approximately 20 yr.). With this thought in mind, the curves for 10,000,000 min. would indicate that raising the temperature above about 80 C. causes a marked increase in the amount of relaxation which takes place. The curves shown in Fig. 17 all refer to copper tested above the yield stress. Very similar curves would be found for copper tested below the yield stress but the remanent stress would be from 5 to 15 per cent higher.

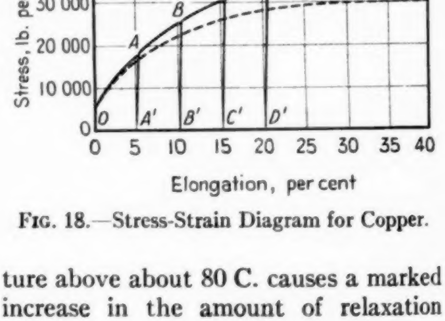

FIG. 18.—Stress-Strain Diagram for Copper.

USE OF THE CURVES

The tests just discussed have been designed to survey the field of relaxation in copper in a somewhat general way. The more important influences of stress, temperature, and amount of cold work upon one another have been investigated and an effort has been made to set forth the results in a useful manner. Care should be exercised in making use of the information contained in the data to make certain that the cases to which it is applied have conditions similar to those under which the tests were made.

In any given application the condition of the copper used should be known or determined from a tension test. A typical stress-strain diagram for copper appears in Fig. 18. The dotted line represents the curve obtained with a test specimen of annealed copper, plotting the load stress (load divided by original area) against the unit elongation in per cent. The full line, *O A B C D*, etc., illustrates the relation between the true stress[6] (load divided by actual area) and the unit elongation. Had the load been removed from the test specimen at any one of the points, *A*, *B*, *C*, and *D*, the resulting unloading curve would be *AA′*, *BB′*, *CC′*, or *DD′*. Subsequent reapplication of the load would show the loading curve *A′A*, *B′B*, *C′C*, or *D′D*, respectively. Suppose that the stress-strain diagram of a sample of copper, whose degree of cold work were unknown, showed a yield stress of say 30,000 lb. per sq. in., it would then be evidence that the sample had had about 15 per cent cold work or its equivalent.

From this information one may determine whether or not the service stresses would be above or below the yield stress. Knowing this, he would be able to estimate from Fig. 15 what remanent stress to expect after, say, 20 yr. at room temperature for any given initial stress. In these curves, the full lines refer to copper loaded above the yield stress and the dotted lines to copper loaded below the yield stress.

For service above room temperature, the data of Fig. 16 may be used, but it should be noted that the curves shown refer to an elapsed time of only 17 hr. It would therefore be better to refer to either Fig. 14 or 17. The latter does not give curves for copper tested below the yield stress, but up to about 180 C. such curves would indicate remanent stresses of from 5 to 15 per cent higher than those shown.

CORRELATION WITH CREEP TESTS

A complete correlation between relaxation and creep data has not as yet been made; however, some relevant considerations may be mentioned. Foremost among the factors concerned is the following question: Is the creep rate at any stress, σ, obtained in a relaxation test made by holding the strain constant at some value, say ϵ_0, the same as that found from a creep test made with a constant stress equal to σ, and with a total strain which has been allowed to proceed until it has become equal to ϵ_0? In other words, will the creep rate for the specimen in Fig. 2 be the same when the stress has been reduced from σ^* to, say, σ_{2-3} as that of a creep test started at 0, with a stress σ_{2-3} and allowed to creep until the total extension is ϵ_0?

If this is not the case, there is the other possibility, that the creep test specimen at stress σ_{2-3} will reach the same creep rate, not at the same strain ϵ_0, but at the same time, t, (line $A''\ 2''\ 3''$, Fig. 2). Although the latter consideration does not seem the more probable, a large amount of work on the correlation of creep and relaxation data indicates that it agrees much better with experiment. Possibly a compromise between two such assumptions may provide the closest agreement.

All experimental evidence seems to show that the creep speeds found in relaxation tests are larger than those obtained from creep tests under what were assumed to be similar conditions. As a result, the magnitude of the de-

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[6] True stress = load stress $\left(1 + \frac{\text{percentage elongation}}{100}\right)$.

crease in stress in relaxation is larger than would at first be expected from considerations based on creep rates observed in tests made with constant stress.

Another phenomenon which is allied with the above statement is illustrated in Fig. 19, which shows that, under certain conditions the change in the creep rate produced by reducing the stress by an amount $\Delta\sigma$ may be quite different from the change caused by increasing the stress in the same amount. The figure shows an interrupted creep test, started with a stress of 12,700 lb. per sq. in. At point *A* the stress was raised to 13,170 lb. per sq. in. and at *B* it was restored to the initial value of 12,700 lb. per sq. in. It will be seen from the figure that the ratio of the creep speeds, $\frac{u_2''}{u_1''}$, for increasing the load was very much larger than the ratio $\frac{u_3''}{u_4''}$, found when the load was decreased by the same amount. Hence in unloading, the creep rate does not seem to alter nearly as much as in loading. This is apparently another manifestation of the fact that large amounts of load must be removed in order to bring a relaxation test specimen back to its "constant" length and thus satisfy the conditions of the test.

Another effect that no doubt is important in relaxation may be described with the aid of Fig. 20. Here the creep curve *AB* has been interrupted at *B* by decreasing the stress and *BC* is the corresponding elastic recovery. If no other influence would exist, creep would then proceed along line *CDE*. However, experiments show that the curve found usually resembles *CD′E′* or *CD″E″*. That is, a certain amount of recovery is superposed on the creep curve *CDE* just after the unloading. In general, the effect of recovery is soon masked by the creep that takes place at the reduced stress. The principal influence caused by recovery following an unloading is that it temporarily slows down or retards the increases of length under creep at the reduced stress.

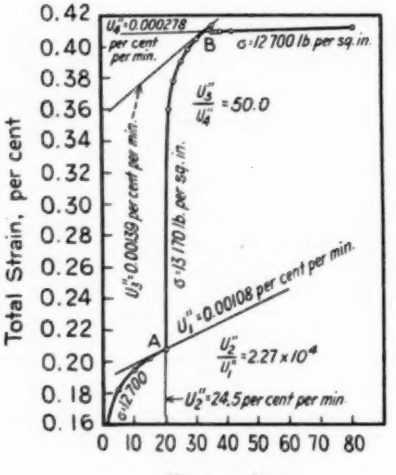

FIG. 19.—Interrupted Creep Test at Room Temperature.

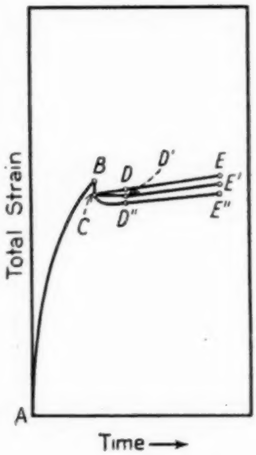

FIG. 20.—Interrupted Creep Test.

When relaxation conditions are approximated by very small steps, recovery seems to be less important. However, the extent to which it effects a relaxation test as it is actually carried out can be noted from Fig. 12. Consider for example the curve for 15.4 per cent copper. In the first part of the test, the machine was unable to follow the load rapidly enough, and had to unload continuously for 3 min. before enough stress was removed to restore the bar to its initial length. Assuming that this decrease in stress was uniform with time, the unloading curve previous to point *B* would look something like *AB*. During this period the load decreased more slowly than the conditions of relaxation would prescribe. Hence the stresses were higher during this time and as a result produced more creep than would have occurred in true relaxation. Therefore, it was necessary to remove more load in order to restore the initial length. This consideration indicates that the true relaxation curve for the early part of the test would look like *CD*. True relaxation conditions are established at *B* but recovery due to the initial unloading causes a slower creep rate from *B* to *D*. After point *D* is reached it is likely that conditions previous to *D* no longer have a great influence. In any event, a good approximation to true conditions of relaxation ought to be obtained beyond point *D*.

Another factor which probably has an appreciable effect on the results of creep and relaxation tests is the history of the material previous to testing. The development of internal stresses during drawing, rolling, stretching, etc., no doubt initiate within the material a process of readjusting itself under these new conditions. The rapidity with which such readjustment takes place probably has much to do with the behavior of the material when subjected to the various mechanical tests and the degree of readjustment that occurs depends upon the manner of stressing and the length of time that the forces are applied. Subsequent removal of the external loads further complicates the picture while a sequence of loadings and unloadings at different speeds defies accurate analysis. Because of such a situation, a sample being tested or being used in service will not alone be effected by the conditions that immediately prevail, but will also be influenced by effects set up in some previous stressing.

From a mechanical point of view, the things to be considered in connection with the previous history will include: the speed of stretching (or cold working), the length of time subjected to load, the amount of time between cold working and testing and the number of reversals of the applied loads.

Tests are now being carried out in an attempt to establish just how important all these varied factors are, and to establish them in their proper relation to one another.

SUMMARY

1. The relaxation of copper at room temperature and probably up to about 200 C. may, for practical purposes, be represented by an expression of the form:

$$\sigma = \sigma^* [1 - A \cdot \log (1 + Bt)]$$

where σ = the stress at any time t,
σ^* = the initial stress, and A and B are constants.

2. A marked increase in relaxation takes place when the temperature is raised above about 80 C. At 150 C. the stress is estimated to fall to about 20 to 25 per cent of its initial value in a service time of 20 yr.

3. Recovery effects tend to decrease the creep rate found in the early part of a relaxation test.

4. The dissimilarity between the speed

laws for decreasing and for increasing stress gives rise to rather large amounts of relaxation.

5. Further creep and relaxation tests are at present under way and it is hoped that they will throw additional light on the subject.

Acknowledgment:

Acknowledgments are due to the following:

The Engineering Foundation for financial support of the investigation during the years 1935 and 1936.

Dean E. A. Holbrook of the University of Pittsburgh for arrangements made through the University and for his interest in the work.

Mr. L. W. Chubb, Director of the Research Laboratories, Westinghouse Electric and Manufacturing Co., for placing the facilities of the research laboratories at the author's disposal and furnishing the means to build the test apparatus.

Dr. A. Nadai for arranging the various details of the program and for his support, advice and influence as the work progressed.

Mr. E. A. Davis for assistance in conducting a large number of the tests and to Messrs. N. Mowat and J. Gianino for help in making the interrupted creep tests.

DISCUSSION

MR. CHARLES C. DAVENPORT[1] (*presented in written form*).—In many industrial applications the stress is not constant with time, but changes gradually as plastic flow occurs; an important special case is relaxation. Almost all of the information available on the plastic flow of metals is in the form of creep curves at constant stress; Mr. Boyd's paper is one of the first to give much information on relaxation. It would be quite useful if "relaxation" could be predicted or derived from the numerous available creep data, but there is no process of logic alone by which this is possible. Several theories have been suggested for this purpose, but which one among them will give the best results can be found only by comparing the theoretically derived relaxation curves with the experimental one. With this object in mind, the writer has made creep and relaxation experiments in torsion, also on copper, at temperatures of 150 to 200 C. but in a much lower stress range than this paper, that is, 500 to 1300 lb. per sq. in. shear stress. These investigations will be presented shortly before the American Society of Mechanical Engineers. It was found that the experimental value of the time to produce a given stress relaxation falls between the value calculated by strain hardening, which gives an upper limit, and the value obtained by time hardening[2] which provides a lower limit. Similar conclusions were reached in this paper.

It is suggested that an effect mentioned by Bailey may explain why the strain hardening theory in the above calculations implies that the metal is stiffer in relaxation than tests indicate. Slip at a point in the metal causes local heating and consequently lowers the resistance at this spot to further slip. It is, therefore, expected that this further flow will not produce as much hardening effect. This result is exaggerated by high flow rates. Now, when a given stress and strain are attained in relaxation, the previous stresses have been larger, and therefore higher flow rates have been present than when the same stress and strain are reached in creep. From this reasoning it is expected that at the same strain the material will be weaker in relaxation than in creep. The strain hardening theory implies equal strength at this point, but tests in the paper and by the writer both showed that the material actually was weaker in relaxation and accordingly had higher flow rates. It was also found that the strain hardening theory gave better results for the lower temperatures than for the higher ones. This may be explained in a similar manner. Since the initial flow rates are smaller at the lower temperatures, the above-mentioned effect has not as much influence.

Since many important industrial problems are concerned with much lower flow rates than those measured either by the writer or reported in the paper, it is believed that with these flow rates

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[1] Graduate School of Engineering, Harvard University, Cambridge, Mass.

[2] Time hardening was used with the correction suggested by the writer in a discussion of the paper "Interpretation of Creep Tests for Machine Design," by C. R. Soderberg, *Transactions*, Am. Soc. Mechanical Engrs., August, 1937, p. 511.

the strain hardening theory will apply still better in the calculation of relaxation from the results of creep, because the above-mentioned effect will have less influence.

The author has used rather high stresses and some high temperatures for copper, so it is to be expected that the effect just mentioned will be rather important in these extreme cases. The writer would be quite interested to know whether the author found any tendency for the tests to check the strain hardening theory better for tests with the lower initial flow rates, which the above suggestion would indicate as probable.

Mr. P. G. McVetty.[3]—There is one point in the history of this development which may be of interest. We had been making tension creep tests for several years when the need for reliable relaxation data became urgent. Since we had available considerable constant stress creep data, we tried very hard to solve the relaxation problem by using existing data to avoid development of new equipment. Up to the present time, attempts to construct the relaxation curve mathematically from the conventional creep curve have not given entirely satisfactory results.

Mr. Boyd has shown that relaxation tests may be made with relatively simple equipment simulating the conditions which exist in flange bolts and similar applications. Mr. Mochel has made relaxation tests at high temperatures at our South Philadelphia Works, using commercial grades of heat-resisting materials, and his results are in close agreement with those of Mr. Boyd. From our experience, it seems desirable to recommend that the solution of relaxation problems be based upon the relaxation type of test rather than the creep test, until a more satisfactory method of correlation of the two tests has been developed.

Mr. John Boyd[4] (*author's closure, by letter*).—Mr. Davenport has asked whether or not the tests show a tendency to check the strain hardening theory better for low initial flow rates than for the higher rates. Since there may be some uncertainty as to just what is implied in a strain hardening theory, an answer to the above question must be somewhat indirect, but it is hoped that the following statement will furnish the information desired.

Observed creep speeds in relaxation have been compared with similar speeds computed from data obtained by creep tests. The computed values depend of course upon what assumptions are made in attempting to secure relaxation data from creep test information. For the present discussion let the creep rate after any unloading such as 2-2′ in Fig. 2 be assumed to be the same as that found in a creep test at stress $\sigma_{2'-3}$ which has been allowed to proceed until it has the same total extention ϵ_0. Using this assumption, an expression may be written which will give the creep rate (for a simulated relaxation test) at any time t. This rate can then be compared with the value, at time t, found in an actual relaxation test having the same initial conditions.

Such a comparison has been made. It is found that the discrepancy between the observed rates and those computed in the above manner is less at 8000 lb. per sq. in. (the lowest value for which comparative data was available) than at, say, 20,000 lb. per sq. in. This is apparently similar to what Mr. Davenport has found. We expect soon to publish additional work on relaxation in which detailed information and comparisons will be furnished.

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[3] Mechanical Engineer, Research Laboratories, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

[4] Engineering Foundation, New York City. Present connections: Research Engineer, Research Laboratories, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

NEW EQUIPMENT FOR CREEP TESTS AT ELEVATED TEMPERATURES

By P. G. McVetty[1]

Synopsis

This paper describes a creep testing machine which provides from 12 to 60 tests at different stresses in the same furnace. The furnace consists of a large cylindrical mass of alloy steel containing twelve vertical holes 30 deg. apart for the test specimens under stress and a larger central hole for pretreatment of specimens without stress. Test specimens are supported on knife edges at the top and loaded by twelve radial levers and weights which hang in an annular space around the foundation. The wall of the furnace, consisting of concentric shells of polished nickel and aluminum with powdered silocel between, rotates on ball bearings to facilitate creep measurements.

Two methods of creep measurement are provided. One consists of comparison rods extending to the top of the furnace from both ends of the 20-in. gage length. The relative vertical displacement of these rods is measured by a dial gage reading to 0.0001 in. Also, the furnace is surrounded by a track on which a specially designed micrometer-microscope carriage moves. This allows measurements on suitably marked platinum targets at the ends of the 20-in. gage length or any intermediate points. The shell carries quartz windows which may be adjusted vertically and the central steel column is slotted to make the reference marks visible. Automatic control of voltage and temperature have been provided and room temperature control may be added.

Introduction

The design of apparatus for creep tests at elevated temperatures has received the attention of many able investigators and we now have dependable equipment and, to a certain extent, standardized procedure[2] which may be expected under suitable conditions to give consistent results. In the use of such equipment to appraise available alloys and to secure data upon which working stresses may be based, a large number of testing units are necessary. Alloys for high-temperature service are being developed much more rapidly than they can be tested with a reasonably extensive installation of the type of equipment now being used.

It has been found that the type of alloys best suited to high-temperature applications have a tendency to depart from the homogeneity and isotropy usually assumed in the commercial use of test data. Also, these alloys are not always stable, either structurally or

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[1] Mechanical Engineer, Research Laboratories, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

[2] Tentative Method of Test for Long-Time (Creep) High-Temperature Tension Tests of Metallic Materials (E 22 – 35 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 1291 (1935); also 1936 Book of A. S. T. M. Tentative Standards, p. 1316.

dimensionally, at the temperatures of their use. As a result, we find "negative creep," anomalous stress-creep relations, and differences in creep curves obtained under essentially identical conditions. No amount of refinement of the details of testing equipment and procedure can produce uniformly consistent test data if appreciable variations exist among the test specimens used.

It is doubtful whether the designers of the type of creep equipment commonly used ever contemplated the necessity of making a hundred or more simultaneous tests. With each test having its own furnace and temperature control a real problem is introduced in the production and calibration of satisfactory furnaces and the maintenance of a large number of control instruments. Another problem results from the possible effect of unavoidable temperature fluctuations in different furnaces upon the comparison of tests of different materials or the same material under different stresses. When anomalous results are obtained, it is difficult to decide whether the discrepancies result from a lack of absolute uniformity among test specimens, or slight variation in testing conditions, or both. The present trend is undoubtedly in the direction of statistical studies in which a relatively large number of similar tests are averaged. Unless this is done, it is necessary to assume a degree of uniformity among test specimens and the details of testing procedure which is practically impossible to realize under present conditions.

In considering installation of equipment for a large number of tests, the solution is not necessarily the duplication of complete individual units even though they may be satisfactory individually or in small groups. Many factors suggest a reconsideration of the fundamental aspects of the problem, even though it may involve a departure from a more or less standardized type of testing equipment. Multiple unit installations are not new.[3,4,5] Among these, the recent development by the General Electric Co.[5] represents a distinct advance toward facilities for extensive creep-testing programs. This subject is treated in greater detail in the section on Development of Equipment.

UNIFORMITY OF MATERIAL

In any discussion of uniformity of material, it is necessary to distinguish between two fundamentally different viewpoints. In the purchase of materials standard specifications must allow a reasonable latitude in the composition. As the permissible variations are reduced, rejections result in increased costs which must be considered in comparison with the advantages gained by more strict specifications. The only fundamentally sound solution is to conduct enough tests to make sure that the properties of the weakest material acceptable under the specification are known. This leads to a statistical study of the material which is not usually impracticable at normal temperatures. At elevated temperatures, the difficulties of statistical studies increase enormously and very little work in this direction has been possible. The logical method of attack is to provide sufficient testing facilities to allow rapid progress on extensive testing programs.

From the standpoint of consistent creep test data, the question of uniformity must be approached in a different way. It is a well-known fact that

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[3] F. H. Norton, "The Creep of Steel at High Temperatures," p. 11, McGraw-Hill Book Co., Inc., New York City (1929).

[4] P. Faber, "The Work of the Turbine Department," *Brown Boveri Review*, Vol. 17, p. 50 (1930).

[5] P. H. Clark and E. L. Robinson, "An Automatic Creep Test Furnace-Guide," *Metals and Alloys*, Vol. 6, p. 46 (1935).

creep properties are very sensitive to slight changes in analysis and structure and they may be affected by sub-microscopic differences in structure and the presence of oxygen, nitrogen or other components not usually shown in the analysis. In making high-temperature tests the usual purpose is to find the relations among four variables—temperature, stress, time and plastic deformation or creep. These relations may be studied for any two of the variables by keeping the other two constant, or nearly so, provided that the test specimens used are alike. It is obviously difficult to study these relations if structural and dimensional changes independent of stress accompany long exposure to elevated temperatures. The problem is complicated further if these uncontrolled variables are different in different specimens. These factors have led to the use of a great amount of care in the selection, heat treatment, machining and final magnetic testing of specimens to ensure the greatest possible degree of uniformity among the test specimens used for creep tests.

When these precautions have been taken to secure reliable data on the properties of a given type of material, it is still necessary to explore by suitable high-temperature tests the effects of variations permitted by the specifications. In this way specifications based on a proper balance between desirable properties and resulting costs are obtained. Under present conditions, this problem can be handled best by large numbers of reliable creep tests with parallel metallographic investigations of structure and careful analysis of the results obtained. The proper proportioning of testing effort to suit the relative commercial importance of a given material is an economic problem requiring individual study of each material.

CREEP-TEST REQUIREMENTS

The fundamental requirements for tension creep tests are relatively simple. It is necessary to provide means of heating the test specimen to the desired temperature with negligible variations throughout its mass. The difficulty of accomplishing this end increases with the length of the specimen and with the ratio of specimen length to furnace length. This temperature must be maintained with negligible fluctuations in the heating and cooling cycle and from day to day over long periods of time. Means must be provided for applying an accurately measured stress uniformly distributed over the cross-section and this must be maintained constant. Finally, it is necessary to measure accurately the dimensional changes which take place in the length of the test section while it is maintained at constant stress and temperature. If these fundamental requirements are met, it becomes possible to study stress-strain-time-temperature relations which should be independent of the details of testing equipment and procedure used to satisfy these requirements.

It is obvious that the importance of each requirement depends upon the manner in which the resulting data are to be used. In many cases, a desirable feature is obtained only by sacrificing some other factor. These compromises occur so frequently that it would be difficult to design any equipment equally acceptable for a wide variety of applications.

The maintenance of a uniform stress over a long period of time is relatively easy. The simplest method uses weights applied directly to the specimen or to a lever system acting on the specimen. For small deformations it is customary to neglect the small increase of stress which accompanies a decrease of cross-section as creep proceeds. For larger deformations, automatic compensation for this stress change[6] may be provided if desired.

In speaking of stress as applied to a test specimen, it is assumed that the stress is uniformly distributed over the cross-section from one end of the gage length to the other. With short specimens and small radius fillets near the ends of the gage length, creep may be increased by stress concentration near the ends. This effect is reduced by proper design and increase in the length of the specimen. Lack of homogeneity of material in the test specimen also causes stress concentration. If present, this factor must be considered in the interpretation of test results.

Whether or not a steady state of stress is desirable is open to argument. In service, each application has its own stress variations ranging from static loading or occasional impact shocks to the cyclic variations accompanying vibration. Duplication of these variations in the creep test would, undoubtedly, have a pronounced effect upon the creep data. Various plans for controlled stress variations have been considered, including axial and transverse vibrations or combinations of the two. The use of an inductive furnace winding gives an axial vibration while the superposition of an unbalanced rotor on the load gives a more complicated stress variation. While these procedures have some advantages, it is doubtful if they outweigh the disadvantage of departure from uniform stress during the test except in special cases.

The maintenance of uniform temperature over long periods of time becomes increasingly difficult as the allowable variation is decreased. Uniformity throughout the test section depends primarily on furnace design while uniformity from day to day depends also upon auxiliary equipment. From any assumption of allowable scattering of points on the creep curve and the coefficient of expansion of the test material, it is possible to compute approximately the corresponding allowable temperature variation. There is some difference of opinion as to whether great care should be used to reduce variations to a minimum or to accept appreciable variations and apply corrections to the observed creep data. On account of the uncertainties which may be introduced by these corrections, the author prefers to avoid them so far as possible.

The accurate measurement of creep may be obtained in various ways. For precise measurements, the Martens type extensometer previously described[7, 8] has given excellent results for many years. Its use requires precise temperature control with the result that readings are obtained very slowly. Our experience indicates that five or six machines are the maximum number that can be operated satisfactorily by one observer if readings are taken daily. Multiplication of units of this type presents a difficult problem of maintenance. The need for this degree of precision results from the extremely small limiting amount of creep in some applications. An allowable creep of 0.01 per cent per year is not uncommon. If the creep rate were constant, this would amount to only about 0.000002 in. per inch per week. Actually, the creep rate is relatively rapid at the start of the test and gradually diminishes in value. This makes it desirable to measure rates considerably smaller than the average.

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[6] J. Cournot and K. Sasagawa, "The Investigation of the Viscosity of Alloys at High Temperatures," *Revue de Metallurgie*, Vol. 22, No. 12, December, 1925, p. 753.

[7] T. D. Lynch, N. L. Mochel and P. G. McVetty, "The Tensile Properties of Metals at High Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 25, Part II, p. 5 (1925).

[8] P. G. McVetty and N. L. Mochel, "Tensile Properties of Stainless Iron and Other Alloys at Elevated Temperatures," *Transactions*, Am. Soc. Steel Treating, Vol. 11, p 73 (1927).

Such measurements are of the same order of magnitude as the distance between atomic planes in alpha iron. The most direct method of improving conditions of measurement is to multiply the quantity to be measured by increasing the gage length.

In other applications, the amount of creep to be measured is greater. This allows the use of short specimens with less precise means of measurement. The wide range in requirements regarding the permissible amount of creep indicates the desirability of considerable latitude in the means used to satisfy creep-test requirements.

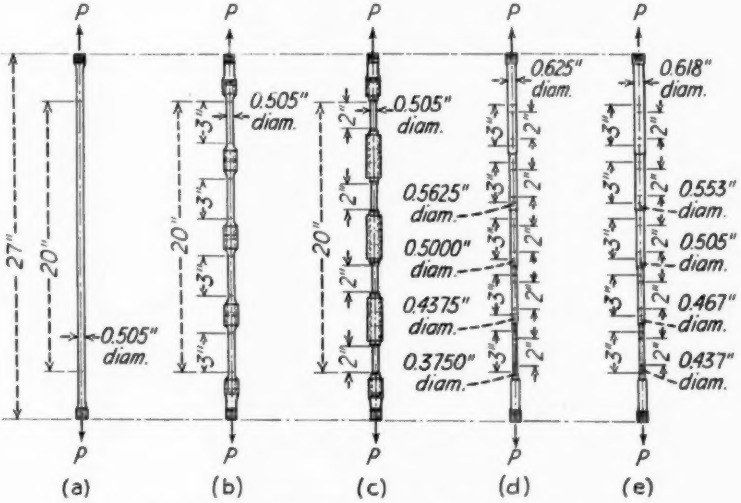

FIG. 1.—Possible Variations in Test Specimens.

DEVELOPMENT OF EQUIPMENT

Test Specimen:

In developing the new equipment, the design of a suitable test specimen received first consideration. Most of our work has been done on specimens 0.505 in. in diameter with a 3.00-in. gage length.[7] The A.S.T.M. tentative standard subsequently developed has the same diameter with a 2.00-in. gage length.[2] Others have used smaller diameters to allow tests at higher stresses without increasing the size of the equipment. There is no reason why a specimen 0.505 in. in diameter is a better representative of a given material than any other cross-section. There is, however, an advantage in standardization for comparison of data from different sources on account of the relation between surface and cross-sectional area. Unless this relation is maintained, creep tests would not be expected to be directly comparable, especially if appreciable scaling occurs. A preliminary study included the preparation of alternative designs for $\frac{3}{8}$- and $\frac{1}{2}$-in. diameter specimens. The reduced size and weight of equipment with the smaller size offered several advantages. On the other hand, a large amount of data has been collected using specimens with a diameter of 0.505 in. It was decided finally to build the larger unit since it could be used equally well for smaller diameter specimens if desired. This eliminates

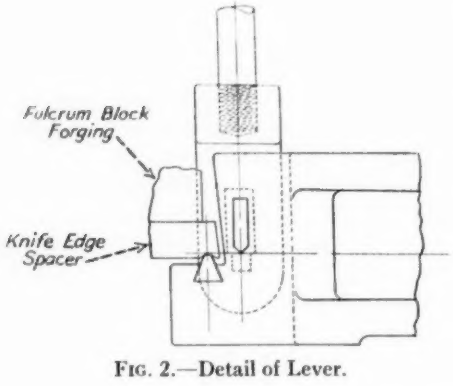

Fig. 2.—Detail of Lever.

at the start the necessity of considering effects of differences in the relations between surface and cross-sectional area in comparing results with creep data already available.

The main objection to a long test specimen has been the difficulty of securing uniform temperature over the increased length. The additional difficulty of securing more test material of uniformity suitable for creep tests is far from negligible. This led to a design in which it is possible to use short or long specimens as desired.

Figure 1(*a*) shows a specimen with a 20-in. gage length. This may be subdivided by suitable reference marks on platinum targets to allow comparison of creep properties at different places along the bar or comparison of different gage lengths on the same bar. By using various combinations of targets, a detailed study of uniformity of the material and effect of different gage lengths may be made on the same bar.

Figure 1(*b*) shows the method of coupling and testing four specimens of the type we have used for the past thirteen years. Figure 1(*c*) shows a similar method applied to the specimens recommended in A.S.T.M. Tentative Method E 22 – 35 T.[2]

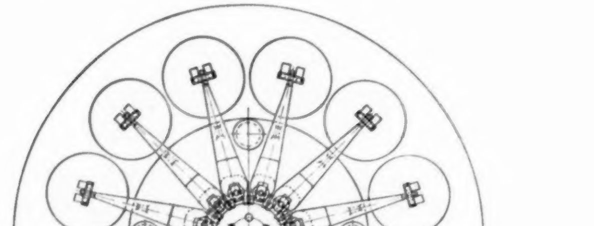

Fig. 3.—Arrangement of Levers, Section *A*-*A*, Fig. 5.

Figure 1(*d*) and (*e*) indicate what can be done with a single multiple specimen to obtain five creep curves at different stresses on the same bar. Figure 1(*d*) shows a uniform change in diameter and Fig. 1(*e*) a uniform change in area and stress. For example, in Fig. 1(*e*), a load of 300 lb. at the end of the 20 to 1 lever will produce stresses of 20,000, 25,000, 30,000, 35,000 and 40,000 lb. per sq. in. The two diameters less than

0.505 in. will give the two higher creep rates which have the least influence in determining creep properties at the lower stresses. This minimizes the objections to a decrease in specimen diameter below 0.505 in. and the corresponding changes in the relation between surface and cross-section.

With this method, any desired relation among the stresses may be used by suitable specification of the individual diameters. The advantages of simultaneous duplicate or triplicate tests of six or four materials in the same furnace at the same time are obvious.

The use of specimens 0.505 in. in diameter makes direct loading impracticable. A convenient lever ratio is 20 to 1 because it gives a ratio of 100 to 1 between applied load and stress on a 0.505-in. diameter specimen. The cylindrical arrangement of the specimens offers the best conditions for uniform temperatures leading to radial arrangement of the levers. Figure 2 shows the design of knife edges in the lever used. Special precautions were taken to make the distance between these knife edges as short as possible because of its effect upon the long arm and the over-all diameter of the machine. Figure 3 shows the radial arrangement of the levers and Fig. 4 the fulcrum block which takes the reaction of the inner ends of the twelve levers.

Construction of Furnace:

Assuming that each test might be loaded to a maximum stress of 50,000 lb. per sq. in., the design indicated a maximum total reaction of 120,000 lb. A structural frame to take this load would interfere with free access to the furnace for creep readings, so heavy alloy steel blocks were used, carefully fitted together to form the main portion of the furnace and to support the load imposed by the twelve tests. These blocks were drilled to take the twelve specimens and slotted for optical creep readings. Figure 5 shows a vertical section of the furnace and the arrangement of the steel blocks.

This method of construction is not without disadvantages. The material used for heat insulation at the ends has to carry the same load as the alloy steel furnace blocks. For this purpose alternate plates of mica and steel with a total thickness of 5 in. were used. Of this amount, 86 per cent is a high grade of amber mica. To bond the laminations together, these blocks were seasoned for 6 hr. at 160 C. under a pressure of 50 tons prior to machining.

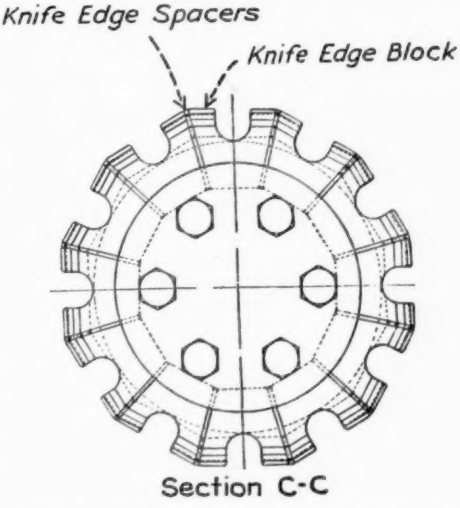

Fig. 4.—Fulcrum Block, Section *C-C*, Fig. 5.

On account of the height of the furnace and the fact that it is built up of several blocks in compression, there is some evidence of lack of stability. This has given no trouble in the first unit but it is considered desirable to balance the loads across any diameter. In two additional units now being built, four of the furnace blocks have been combined into one and a special grade of porcelain will

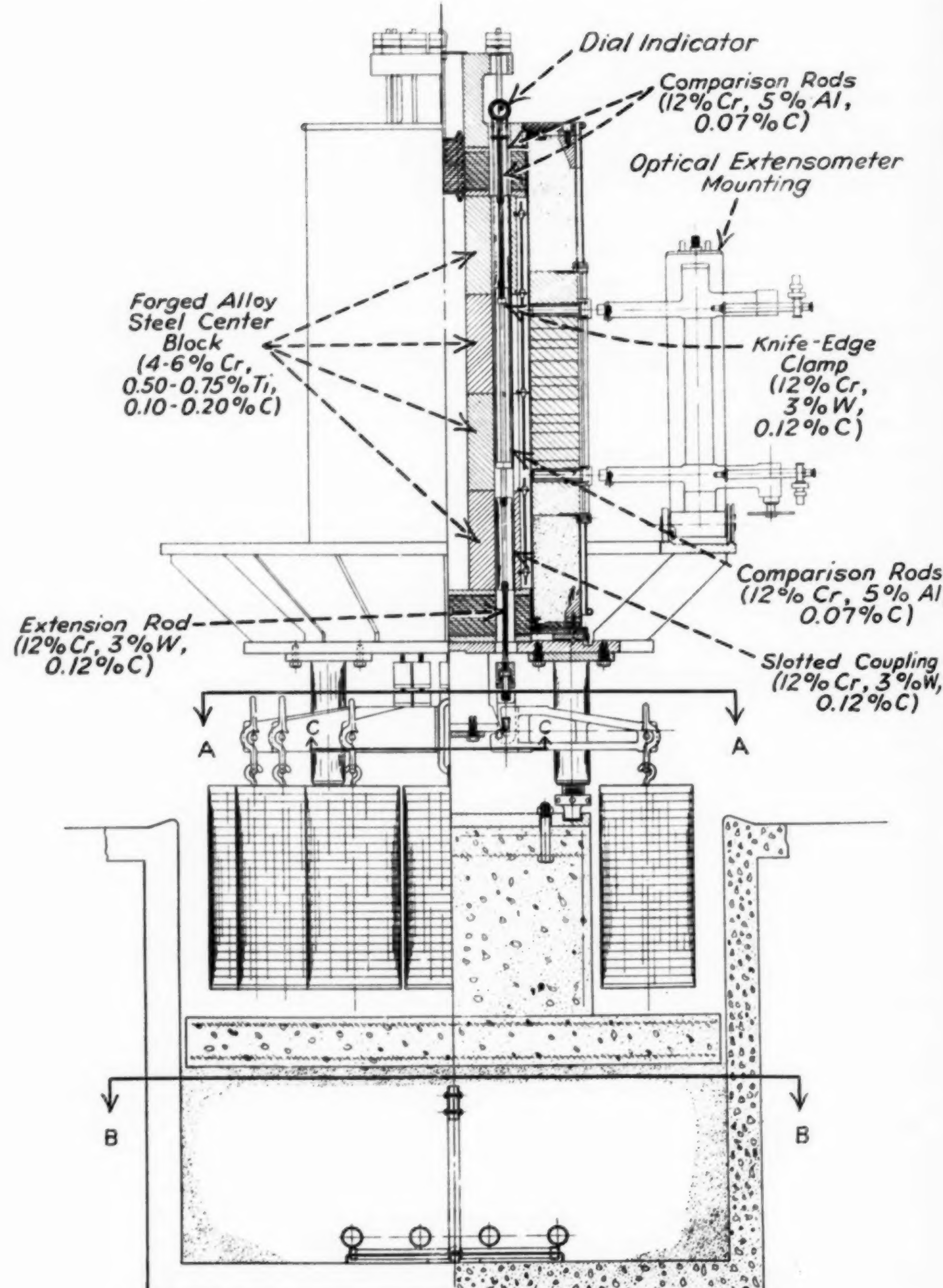
Dial Indicator
Comparison Rods (12% Cr, 5% Al, 0.07% C)
Optical Extensometer Mounting
Forged Alloy Steel Center Block (4-6% Cr, 0.50-0.75% Ti, 0.10-0.20% C)
Knife-Edge Clamp (12% Cr, 3% W, 0.12% C)
Comparison Rods (12% Cr, 5% Al, 0.07% C)
Extension Rod (12% Cr, 3% W, 0.12% C)
Slotted Coupling (12% Cr, 3% W, 0.12% C)
A
C
C
A
B
B

Fig. 5.—General Assembly of Creep Testing Machine.

replace the mica at the ends. These changes were made to improve stability and to allow greater latitude in the distribution of test stresses.

The question of structural and dimensional stability of materials at operating temperatures has been a matter of serious concern in recent creep tests. There is much evidence to indicate that long exposure to high temperature without stress might be a desirable pretreatment for creep test specimens. To allow this, an axial hole, 5 in. in diameter extending from bottom to top of the furnace, has been provided. One or more lots of specimens may be suspended in this hole for any desired time prior to their use for creep tests. In this way periodical over-all measurements of length may be made as a check on dimensional stability. If this pretreatment produces warping, it will reveal the necessity of studies of non-uniform internal stresses which might otherwise escape detection.

Except for the narrow slot for creep measurements, each specimen is surrounded by a large mass of alloy steel to provide the best possible conditions for temperature uniformity. Since the heat conduction to the ends is necessarily large, the couplings which connect the specimens to the extension rods passing out of the furnace are only slightly smaller in diameter than the holes through which they pass. This serves to facilitate conduction of heat from the furnace to the ends of the specimens and improves the vertical temperature gradients.

Heating Elements and Temperature Control:

Three separate windings are provided to supply heat to the furnace. Because of the experimental nature of the first unit it was impossible to make an accurate estimate of the proper distribution of heat in these windings. Heavy nichrome wire was used at the two ends with a lighter wire in the center. Since the bottom winding would normally require the most heat it was connected directly across the line while external rheostats were provided in the other two sections. The wiring diagram for the furnace is shown in Fig. 6.

In each section the winding is supported by porcelain bushings in nichrome segments. To avoid interference with the slots provided for creep readings, the wire passes up and down between slots in the center section. A similar zig-zag construction is used at the two ends. This type of construction provides a non-inductive winding which avoids objections to possible effects of induction on creep readings.

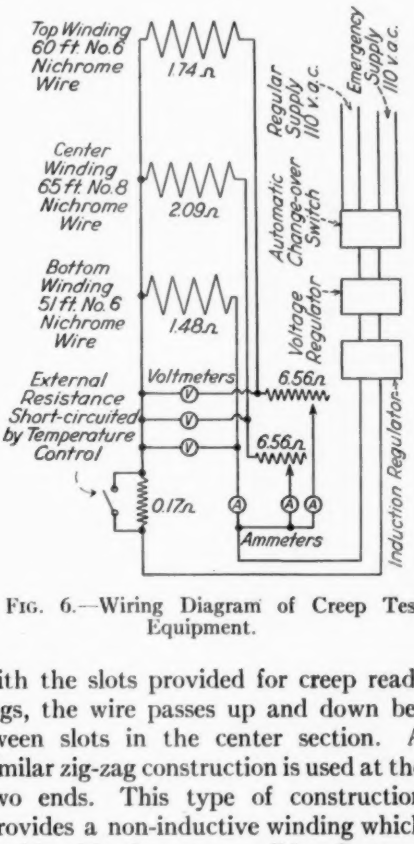

Fig. 6.—Wiring Diagram of Creep Test Equipment.

Serious consideration was given to the possibility of mounting the windings in a separate cage with vertical adjustment for control of heat distribution vertically and continuous rotation to improve circumferential temperature distribution. This was not adopted for the first unit but it may be applied later if a greater degree of temperature uniformity is found to be desirable.

To ensure continuity of operation, two independent sources of electric power have been provided. An automatic change-over switch connects the furnace to the other line in the event of failure of either source of supply.

An induction regulator provides a means of adjusting the furnace current to give any desired temperature up to 1000 F. An automatic voltage regulator helps to maintain a constant heat supply.

A temperature control of the photoelectric type operates a contactor which short circuits a resistance in series with the three furnace windings. This gives temperature control with minimum disturbance of the distribution of heat in the furnace.

A large number of thermocouples are brought out of the furnace to allow studies of circumferential and axial temperature distribution. Number 22 chromel and alumel wires calibrated by the National Bureau of Standards are used. It has been found by experiment that a control couple bedded in the wall of the furnace near the hottest part of the winding gives the best temperature control. This type of control does not provide fully for seasonal temperature variations. On this account, room temperature control is being installed.

In designing the cylindrical insulating shell of the furnace, the original plan called for 24 quartz windows adjustable vertically for creep readings. Because of the large heat loss to be expected from this construction, it was decided to use only two windows and mount the shell on ball bearings so that the windows could be aligned with any one of the twelve tests. The shell consists of a nickel wall adjacent to the winding and two outer aluminum walls. Between the nickel and the first aluminum wall, a radial distance of $5\frac{1}{2}$ in. is packed with powdered silocel. A $\frac{3}{4}$-in air space open at the top and bottom is provided between the two aluminum walls.

Before deciding upon the material and finish to be used for the outer walls, tests were made to determine the relative amounts of radiant heat transmitted. Of the several materials considered, a highly polished cold-rolled aluminum sheet gave the best combination of properties, considering stiffness and weight in addition to relative heat transmission. In comparison with copper, the ease of maintaining a highly polished surface also favored the use of aluminum.

In the original installation, no provision was made for continuous rotation of the furnace shell. After a number of temperature surveys showed circumferential temperature differences of as much as 10 deg. Fahr., a motor drive was installed to rotate the shell at approximately one revolution per hour. This reduced circumferential variation to 3 deg. Fahr. with a corresponding improvement in the scatter of creep readings. The driving mechanism shown in Fig. 7 consists of a motor and reduction gear engaging the large gear at the base of the furnace shell. Provision is made to disengage this drive to allow free rotation of the shell when creep observations are made.

Creep Measurements:

One of the most important factors in creep testing is the reliable measurement of creep after all possible precautions have been taken to minimize the effects of stress and temperature variations and other disturbing influences. In this installation, two independent methods of measurement were provided in addition

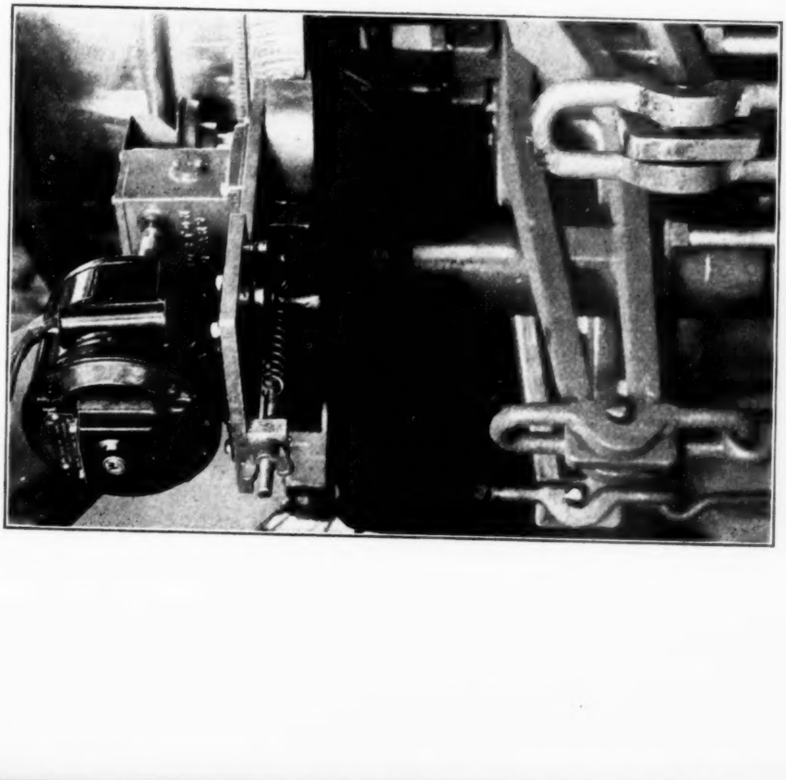

Fig. 7.—Furnace Shell-Drive Mechanism.

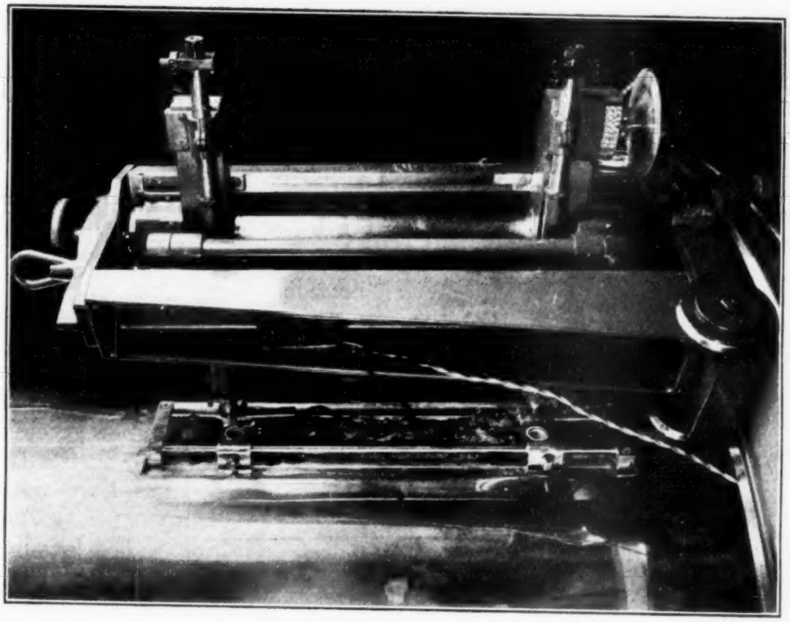

Fig. 8.—Optical Extensometer.

to a possible check of over-all length before and after tests. The first uses measuring or comparison rods extending from the ends of the 20-in. gage length to a point just outside the furnace. A special dial gage applied to these in pairs gives a measure of total change in the 20-in. gage length.

The second method uses a combination of telescopes and micrometer microscopes sighting through quartz windows in the wall of the furnace upon suitably engraved platinum targets located at any desired positions along the 20-in. gage length of the specimens.

For the dial-gage measurements, split knife-edge clamps of chromium-tungsten steel are held at the ends of the 20-in. gage length by nichrome bolts. To each of these are welded two $\frac{3}{8}$-in. diameter rods of an annealed ferritic chromium-aluminum steel chosen for its dimensional stability. These rods extend just outside the furnace at the top. A dial gage with jeweled bearings reading to 0.0001 in. and provided with suitable guides for location on the comparison rods is used for the creep measurements. A special fixture is used for setting the zero of the dial gage.

In the use of this method there was some question about the error which might be introduced by bending of the specimen. While the long length of the specimen and the loose threaded joints between it and the couplings and extension rods through which stress is applied, practically eliminate any possibility of bending, means of detecting bending were provided. These consist of two rods each from top and bottom so that two sets of readings may be taken. This precaution reduces the chance for errors in readings beside offering a continuous check upon transverse deformation of the specimen. Another error may be introduced by dimensional changes in the comparison rods due to long exposure to elevated temperature. Although the material used was chosen for its stability, two checks were provided. Test specimens of the comparison rod material were machined for studies of dimensional stability after various times of exposure to the test temperature. Another check is available since each test has two independent methods of creep measurement over the 20-in. gage length. It is considered also that these rods will approach dimensional stability in service so that any initial error will decrease with time.

The optical extensometer was developed to meet unusually difficult conditions of measurement. So far as the author is aware, precision creep measurements at 1000 F. through a furnace wall of this thickness have not been attempted previously.

Our specifications called for the ability to measure to an accuracy of 0.00001 in., any length between 2 and 20 in. on any one of twelve specimens located 30 deg. apart. The major difficulty in making such measurements results from the necessity of making observations through windows in the furnace shell which prevents the objective from being located closer than $10\frac{1}{2}$ in. from the reference marks on the specimen. To minimize the effect of room temperature changes upon the measuring apparatus, we specified that provision must be made for comparison of readings with a suitably engraved reference standard of low expansion glass in a position protected from the heat of the furnace. Figure 8 shows the extensometer designed and built by a telescope builder to meet our specifications. The heavy frame is made of carefully annealed castings mounted on four ball-bearing wheels. The two outer wheels are grooved to conform with the raised V surface of the track. The two inner wheels have a convex surface which rests on the flat inner ring of the track. One wheel is mounted on an adjustable arm to ensure proper distribution of the weight of the apparatus. A clamp and tangent screws are provided for fine circumferential adjustments.

The upper and lower optical systems are adjustably mounted on a large tube attached to a frame which is free to move vertically in dovetail slides. The reference scale swings on a vertical axis into the plane of focus of the microscopes for checking or out of the line of vision when creep measurements are made. This whole assembly is counter balanced to provide easy vertical adjustment.

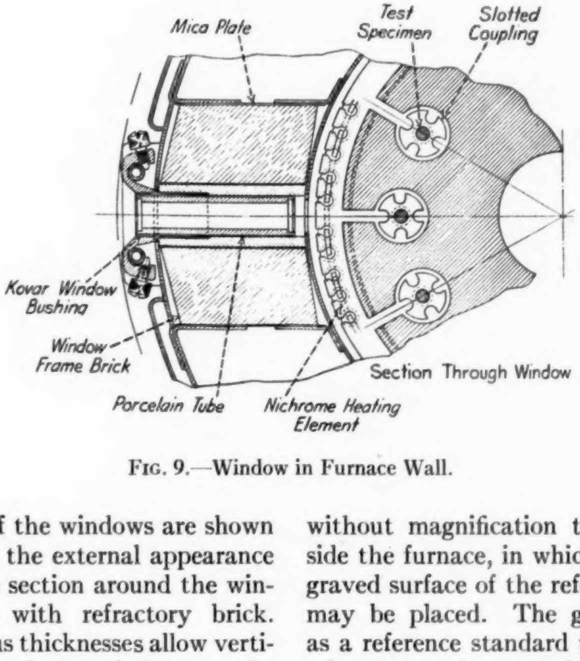

Fig. 9.—Window in Furnace Wall.

The details of the windows are shown in Fig. 9 and the external appearance in Fig. 8. The section around the windows is lined with refractory brick. Blocks of various thicknesses allow vertical adjustment of the windows as desired. The inner window is made of fused quartz ground and polished optically flat and parallel. The outer window is made of optical glass similarly ground and polished. To reduce the effects of air currents between the windows, this space is partially evacuated by continuous connection with a vacuum pump.

The targets on the specimen consist of small pieces of sheet platinum attached by the ignitron welder and carrying engraved reference marks. These marks are lighted by vertical illuminators built into the lens systems. The inside half of the first lens system is located permanently at a distance equal to its focal length from the face of the glass scale. The outside half, having the same focal length, is adjustable to allow its setting at a distance equal to its focal length from the target on the specimen. This arrangement brings a reversed image of the reference mark without magnification to a plane outside the furnace, in which plane the engraved surface of the reference standard may be placed. The glass scale used as a reference standard upon which the micrometer microscope is focused may be swung out of the way to bring the image of the reference mark into the field of the microscope.

In using this apparatus, the arms carrying the upper and lower optical systems are adjusted to a distance apart equal to the gage length to be measured. Vertical illuminators on the glass scale facilitate this adjustment. The arms are then locked to the large diameter steel tube to which they are attached. The frame carrying this tube is then moved vertically in a dovetail slide until the upper microscope is aligned with the upper mark on the specimen. The lower micrometer microscope is then adjusted to the lower mark on the specimen and the departure from the setting on the glass scale is measured. The pitch of the micrometer screw is 0.01 in. and the disk is graduated to 0.00005 in. Readings to 0.00001 in. are obtained by estimation. Both optical systems are provided with filar eyepieces, but these are not ordinarily used because of the slight variation in distance to the specimen in the various positions of the measuring apparatus.

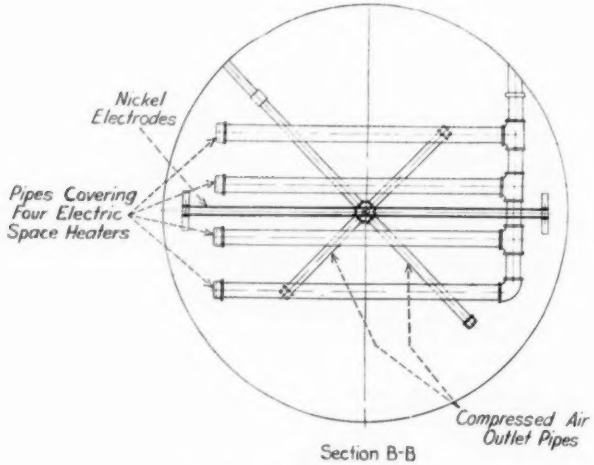
Nickel Electrodes
Pipes Covering Four Electric Space Heaters
Compressed Air Outlet Pipes
Section B-B

Fig. 10.—Foundation Details.

The track on which the optical extensometer travels is a heavy welded structure which was carefully annealed before and after rough machining and then finally machined. The spigot fit for the central column of the furnace, the groove for the ball bearings on which the insulating shell rotates, and the one flat and two angular surfaces on which the optical extensometer travels were all machined at the same setting. The track was finished by grinding and hand-honing until the maximum circumferential variation on any of the three surfaces did not exceed 0.0001 in.

To compensate for any settling of the foundation which would disturb the desired level setting of the track, the entire equipment is mounted on four legs with screw adjustment. These rest on phosphor-bronze disks recessed into the top plate of the foundation. The use of four instead of three legs results from the desire to improve the stability of the structure even though there may be some difficulty in securing equalization of load distribution. Our experience during the first year of operation of the first unit has shown no reason to change this method of support in building the later units.

FOUNDATION

The type of foundation required for this equipment was determined primarily by the optical extensometer. The effects of vibration upon optical measurements are well known. It is suspected also that vibration accelerates creep. In the mounting of telescopes, a dry sand foundation has been found to be quite effective in minimizing the effects of vibration. On that account, this type of foundation was adopted with several precautions to keep the sand dry in service. A vertical section of the foundation is shown in Fig. 5 and details in Fig. 10.

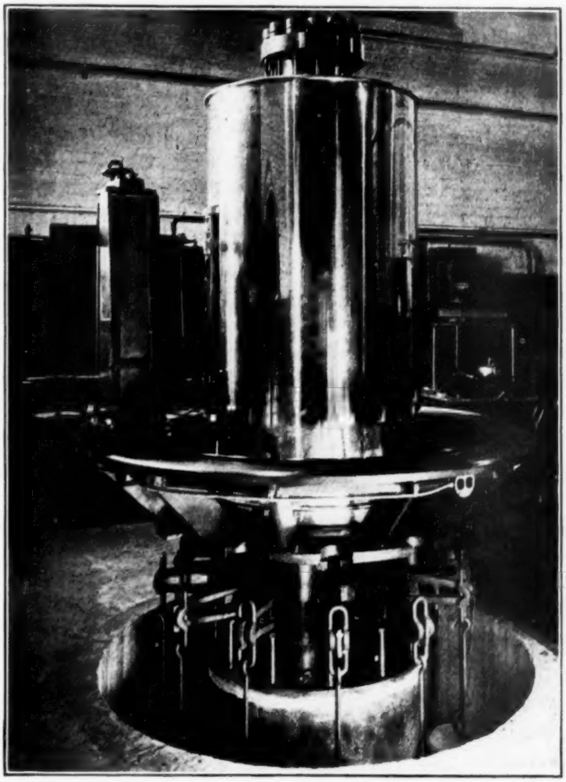

FIG. 11.—Creep Testing Equipment.

To locate the machine at the proper height for convenience of readings, a pit was dug 6 ft. in diameter and 5 ft., 3 in. deep. This pit was lined with a waterproofed wall and floor resting on cinders for drainage. The possible accidental flooding of the pit from the top necessitated some means of drying the sand without disturbing the machine. For this purpose, 5 kw. of electric space heaters were distributed in iron pipes near the bottom with provision to bring the wires supplying current through the wall. On account of the low heat conductivity of sand, a suitably trapped high-pressure air line was connected to pipes under the heaters. These were drilled with seventy-six $\frac{1}{4}$-in. holes 1 in. apart covered with 60-mesh copper screen. Thermocouples were attached to the pipes carrying the heaters to prevent overheating. The pit was then filled to a depth of 2 ft. with carefully dried and graded sand. Near the top of the sand were placed two nickel electrodes, 4 in. wide and 1 inch. apart, extending across the diameter of the pit. Two high-tension leads were brought out to allow measurement of sand resistance with a "Megger." This provided an easy means of determining the condition of the sand at any time. Resting on this sand is a reinforced concrete slab and block extending to the floor level. This provides an annular space below the floor level for the weights and weight carriers. The general appearance of the first unit installed is shown in Fig. 11. The over-all dimensions are 8 ft. in height above floor level and 7 ft., 4 in. in diameter.

Installation and Calibration

The assembly of the equipment was completed and heating started on April 20, 1936. On the first heating, temperatures varied from 645 to 1050 F. in various parts of the furnace. On account of the high heat capacity, changes to effect more uniform heating had to be followed by long waiting for equilibrium conditions. It required several weeks to attain uniform temperatures near 1000 F., the maximum for which the furnace was designed.

The temperature was then reduced to 932 F. (500 C.) and adjustments made to secure the best possible heat distribution at that temperature. These initial adjustments were made with a low stress on dummy test specimens. During this time N. L. Mochel had developed a technique for machining these long specimens and the first lot of nickel-chromium-molybdenum steel was installed on July 17. Adjustments and loading were completed and the first tests started on July 24, 1936.

Operating Technique

The details of operation have been in a state of flux to a certain extent. It was decided at the start that the best way to develop the proper testing procedure was by the conduct of actual tests. This necessarily imposes conflicting restrictions. It is generally desirable to avoid any changes in equipment or methods during creep tests. On the other hand, development requires such changes. As a result of combining the two functions, development has not been as rapid as it might have been. Also, certain allowances must be made in interpreting the test data for changes in method made from time to time.

While considerable development work was done on the use of the optical extensometer we are not prepared as yet to give data obtained by its use. Creep data which have been reported were obtained by use of a dial gage measuring over a 20-in. gage length. Proof as to the extent of influence of possible dimensional instability of the comparison rods will be considered later among the results obtained by the optical extensometer.

The most important influence upon the measurement of creep is the uniformity of temperature throughout the gage length of each specimen and from beginning to end of test. A detailed study of this problem indicates that the apparent results obtained depend upon the number of points of measurement, the location of the thermocouples used, and the circumstances surrounding the measurements. A detailed discussion of these points is beyond the scope of this paper.

In general, chromel-alumel thermocouples are bedded into the surface of the steel blocks forming the furnace at the top, center and bottom of the 20-in. gage length, the middle of the upper and lower windings, and the extreme top and bottom adjacent to the mica insulating blocks. Two additional couples are located in the vertical center at 120 deg.

from those indicating the vertical temperature distribution. Since it was found that best temperature control resulted from connection of the control instrument to the couple nearest the center of the lower winding, additional couples were inserted in this location for spares. Six other couples are clamped to the top and bottom of the gage length of three specimens located 120 deg. apart to give actual specimen temperatures. By means of suitable switches, any of these couples may be connected to a potentiometer sensitive to changes of one microvolt. Any six may be connected at one time to a recording instrument for the study of temperature fluctuations. This gives a continuous record of the principal temperatures.

The final measurement of temperature variations has to be done under conditions which are not the best for obtaining true temperature measurements. Each time that creep readings are taken, thermocouples are inserted near the upper and lower windows and at the center of the gage length. These are held in contact with the surface of the specimen by means of external springs. After insertion the measured temperature rises with the heating of the couple. At the same time, the specimen nearest the windows shows a slight decrease in temperature due to the presence of the window. As a result, the measured temperature rises and then falls, the maximum reading being taken as the desired temperature. This gives three observations on each specimen at least five times a week when creep readings are taken, in addition to the six couples clamped to specimens and the six connected to the recorder.

Cold junctions are maintained at 32 F. (0 C.) in melting ice in a vacuum bottle. To minimize the effects of possible aging of couples, all of those permanently fixed in the furnace are replaced at the end of each run.

In the use of these data, it is assumed that the best measurement of specimen temperature is obtained from the couples clamped to the specimens. It is assumed also, that the best measure of temperature variation is obtained from the couples inserted periodically. It should be noted that couples inserted in this way tend to accentuate variations on account of the impossibility of duplicating contact conditions, and the fact that couples break and are replaced occasionally. Each day, the readings of the six couples clamped to the specimens are averaged and compared with the average of the adjacent 6 readings from the inserted couples. The difference between these two averages is added as a correction for window cooling effect to the readings of each of the inserted couples.

PERFORMANCE

The first series of tests was continued till November 11, a total of 2660 hr., when the furnace was allowed to cool for inspection of the winding and replacement of thermocouples. For this purpose the shell was removed by the overhead crane. After minor repairs, the shell was replaced, furnace heated and the next lot of specimens installed on November 20. The second run started on November 24 and it had continued for 3594 hr. on April 23, 1937. It is interesting to note that after the first test was started the only interruption to actual creep testing was a period of 13 days for cooling, inspection, repairs, heating and starting the second run.

This non-productive time for the first nine months of operation amounts to 5 per cent. Whether or not this can be improved depends upon the extent of repairs and replacements which may be necessary. To anticipate possible trouble with the winding, a new design has been adopted for the later units.

This will allow relatively rapid replacements in the event of winding failure. Under ordinary conditions, the future productive time is estimated to be about 95 per cent.

Since the second run is not yet completed, it is necessary to give results from the first run as an example of the type of data obtainable. Figure 12 shows creep curves from tests at 932 F. (500 C). and stresses of 8000, 12,000, 20,000 and 30,000 lb. per sq. in. These are not representative of the best results obtained since they were affected by a number of changes in adjustments and other minor details essential to the development of the equipment. The change to continuous rotation of the furnace shell, made during the second run also resulted in a decided improvement in temperature uniformity and in the scatter of test points.

The material represented by these curves was cut from a forging prepared and heat-treated by N. L. Mochel. The analysis is as follows:

Carbon, per cent	0.35
Manganese, per cent	0.60
Phosphorus, per cent	0.039
Sulfur, per cent	0.030
Silicon, per cent	0.20
Nickel, per cent	2.70
Chromium, per cent	0.36
Molybdenum, per cent	0.33

In the preparation of this material, a 19-in. octagon fluted ingot was punched and mandrel-forged to a hollow cylinder 18-in. outside diameter by 16-in. inside diameter by 30 in. long. This was given the following heat treatments:

1750 F.—5 hr.—air cooled
1650 F.—5 hr.—air cooled
1550 F.—5 hr.—air cooled
1250 F.—5 hr.—slow cooled
1160 F.—3 hr.—slow cooled
Rough machined
1050 F.—5 hr.—slow cooled

A discussion of the various methods of interpretation used to appraise materials and to determine safe working stresses for high-temperature service is beyond the scope of this paper. A few representative references[9] serve to indicate the trend of thought in the direction of application of creep test data to problems of design and selection of materials.

Conclusions

As a result of this development it appears that reliable creep test data are obtainable by meeting the fundamental requirements of similar test specimens, constant stress, constant and uniform temperature, and means of creep measurement suited to the purpose of the tests. Under these conditions it should not be necessary to specify closely all the details of equipment and procedure in order to secure comparable test data from different sources. That the quality and quantity of data obtained during

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[9] R. W. Bailey and A. M. Roberts, "Testing of Materials for Service in High Temperature Steam Plant," *Proceedings*, Inst. Mechanical Engrs. (British), Vol. 122, p. 209 (1932).

A. Nadai, "The Creep of Metals," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 55, APM-55-10, p. 61 (1933).

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the first year have justified the installation is indicated by the decision to build two more similar units which are now nearing completion. These will permit twelve simultaneous tests at each of three temperatures using a 20-in. gage length. The development of the optical extensometer has reached the stage where it appears safe to predict for the near future that 60 tests on 3-in. gage lengths will be obtainable in each furnace. Up to the present time, comparative tests using the same material in our new and old equipment[7] have not been made. Such a comparison is included in our testing program together with a large number of other investigations of creep properties of various materials under various conditions.

Acknowledgments:

The author wishes to acknowledge the support of the South Philadelphia Works of the Westinghouse Electric and Manufacturing Co., particularly Mr. C. R. Soderberg, Manager of the Turbine Division. He also desires to express his appreciation to Mr. N. L. Mochel, Metallurgical Engineer for selection and treatment of materials and development of a satisfactory method of machining the long specimens, to Dr. L. W. Chubb, Director of Research Laboratories and other members of the Westinghouse organization for many helpful suggestions, to Mr. Ben Cametti for a large part of the design details, and to Mr. K. R. Waugh for the many details connected with the installation, calibration, maintenance and test observations.

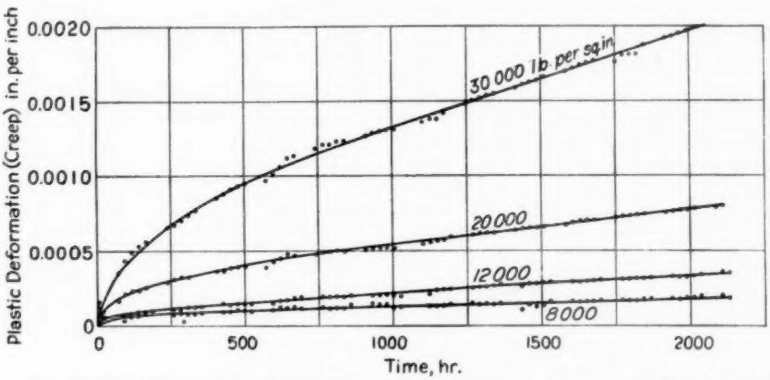

Fig. 12.—Creep Curves for Nickel-Chromium-Molybdenum Steel at 932 F. (500 C.).

DISCUSSION

Mr. Ernest L. Robinson[1] (*presented in written form*).—This paper describes a type of furnace admirably adapted to the requirements of a turbine manufacturer. The measurement of small amounts of extension is provided for, at the same time permitting the simultaneous conduct of a large number of tests. This equipment should prove capable of yielding a large bulk of accurate results.

The designer has shown great courage in his attack on the possibility of multiple gage length specimens. The General Electric Co. has made use of a specimen with two sections, one 0.505 in. in diameter and the other 0.357 in. in diameter, with an extra gage rod where the section changes so as to permit obtaining extension results on two stresses with a single specimen.

For a multiplicity of small gage lengths, Mr. McVetty proposed to use microscopes mounted outside. Not only does the short gage length mean a greatly reduced absolute length to measure, but the setting and reading of the microscopes is likely to be less accurate. In fact, reliable readings of elastic extension are likely to be quite difficult in view of the time element involved as well as the small quantities to be measured. In addition to the difficulty of identifying sufficiently small gage marks, there is also the lack of thermal compensation. However, Mr. McVetty has spared no effort in trying to secure the most precise measuring arrangement possible within the limitations of the method.

The microscope attachment will be ideal for the conduct of creep tests where it is not necessary to measure very small amounts of creep and will facilitate the accumulation of a bulk of results per furnace which is very important. At the same time Mr. McVetty is equipped to use a full 20-in. compensated gage length with a direct reading dial for the measurement of small extensions whether elastic or plastic.

In using multiple-section specimens it may be useful to bear in mind that the percentage of creep in the lower stressed gage lengths will be much less than in the higher stressed specimens. For instance, to have fairly equal absolute extensions to measure, the gage lengths should be increased roughly as the fifth power of the cross-sectional area (tenth power of the diameter), or, putting the relation another way, the gage lengths of the largest diameters in specimens (*d*) and (*e*) of Fig. 1 of the paper might appropriately be made 10 or 20 times as long as the gage lengths of the smallest diameters.

Mr. H. J. Gough.[2]—I should like to ask Mr. McVetty a question essentially concerning the economics of the machine described. The underlying policy involved in the use of a new type of machine is also of especial interest to those of us who have to weigh in advance all the factors involved in such departures

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[1] Turbine Engineering Dept., General Electric Co., Schenectady, N. Y.

[2] Superintendent, Engineering Dept., National Physical Laboratory, Teddington, Middlesex, England.

from previous practice. In my experience with a great variety of kinds of testing there are two cost aspects involved which are not necessarily related and whose combined effect on the total cost of the investigation must be seriously considered with regard to the policy involved: the first is the total cost of the apparatus, including all drawing office and workshop charges; the second is the cost of the actual testing which mainly concerns the staff time involved. In a long-range investigation, the latter usually assumes the greater proportion of the whole and, hence, controls the average cost per test. Now, in my own laboratory, we adopt the principle of a number—forty and more—of individual units. Mr. McVetty's machine is of the multiple unit type, taking up to 12 specimens at a time. In the first place, it does not appear to me that there is any reason to assume that the time involved in making measurements on 12 specimens in either type of machine would differ appreciably; if this is so, the multiple unit machine possesses no economic advantages. It is very desirable, in time-consuming tests of the "creep" type, that the method of test adopted should permit of the utmost elasticity of amendment of any test program as originally drawn up; amended test conditions, such as stress and temperature, may be required in view of the results obtained and such amendments would not necessarily apply to *all* the specimens of a batch numbering as many as 12. Such alterations obviously can easily be made when single units are employed without interfering with any of the other tests; each test is quite independent of the others which are in current progress. But, in the case of the multiple unit described, it will not be so easy and it appears to me that this disadvantage is inherent in such apparatus and will be shown in the testing costs. No doubt this important point has been fully considered by Mr. McVetty and I shall be very glad to have his remarks and experience.

MR. P. G. MCVETTY[3] (*author's closure*).—In preparing this design, it was necessary to keep in mind the limitations imposed by our needs and to choose methods best suited to our objectives. We wanted to provide means of securing data in large quantities without sacrificing the accuracy and reliability of carefully conducted individual tests. To attain this end we drew freely from the experience of other laboratories as well as our own. In many cases, a compromise enabled us to correlate conflicting requirements without departing materially from the desired objectives.

Mr. Robinson mentions the difficulties involved in the use of micrometer microscopes to measure relatively short gage lengths. It was only after very careful consideration of the many factors involved that we departed from the use of the Martens type extensometer which has given us consistently good results for many years. The use of the optical comparator is the accepted method for checking line standards of length and for the finest tool room measurements. In adapting this principle to use at high temperatures we were guided by the fact that other laboratories are using micrometer microscopes for creep measurements. We found by experiment that suitable reference marks on platinum targets gave no difficulty in identification, even after long exposure to high temperature.

We considered carefully the matter of thermal compensation and made provision for it. Any measurement represents some comparison of the object

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[3] Mechanical Engineer, Research Laboratories, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

measured with a reference standard. In the dial gage method the reference standard is a rod within the furnace. Unless this rod is of the same material as the test specimen, departures from constant uniform temperature introduce errors difficult to correct. Also, the matter of dimensional stability of these rods at the test temperature cannot be ignored. We prefer to use as a reference standard a low-expansion glass bar subject only to the effect of room temperature fluctuations which can be reduced to a negligible amount. The graduations on this bar may be checked by Johansson blocks or the blocks may be used directly.

In reference to gage length, the practice in different laboratories varies from 2 in. to 20 in. The 2-in. length requires extremely sensitive and accurate measuring equipment, but most of the laboratories in this country are using it. As the gage length is increased, the creep becomes more easily measured and the less sensitive dial gage method becomes suitable. We use this as a check on the creep of the 20-in. gage length. As a further precaution we use two sets of comparison rods to detect bending of the specimen. The main objections to long specimens lie in the difficulty of securing greater quantities of uniform test material and maintaining uniform temperature over the longer gage length. The varying importance of these considerations in different applications suggests some flexibility in the choice of gage length. In this design, means are provided for measuring various lengths of the same specimen without change in testing conditions.

Mr. Robinson's suggestion of a change from the commonly used length-diameter ratio to one in which the gage length varies as the tenth power of the diameter could not be adopted without a study of its effects. The advantage of approximately equal extensions is probably less important than the relative effect of scaling on test results obtained with various surface-volume ratios.

The question of economics raised by Mr. Gough is extremely important and we found it desirable to study the relations between initial and operating costs. We have had considerable experience with individual unit machines over a period of about fourteen years. In developing the new design, we compared the individual and multiple unit types when applied to installations of various sizes. Only in the case of relatively few tests was the element of first cost in favor of individual units. We could not build twelve single machines for the cost of one of these large units. If we succeed in making sixty tests in one machine, the advantage of the new type is correspondingly increased. From the standpoint of operating costs, our experience with both types indicates a decided advantage in favor of the multiple-unit machine. We have found that daily readings on five single machines require the full time of one man. A large part of this time is used in bringing the furnaces to temperature equilibrium at the exact test temperature. In the new design, the large mass of alloy steel forming the body of the furnace helps to maintain a constant uniform temperature and a man can take readings on twelve tests in fifty minutes. Since labor is the largest element of operating cost this indicates a factor of about ten to one in favor of the multiple unit machine.

In reference to the effect of changes in individual tests upon adjacent tests, our greatest difficulty has resulted from changes in heat conductivity of the alternate plates of mica and steel at the top and bottom. On this account, stress changes disturbed the temperature uniformity within the furnace so that changes in heat distribution among the three windings were required. In the later units, this has been improved by substituting solid porcelain blocks at the ends and a single alloy steel block to form the body of the furnace. With this construction we expect to be able to change tests without difficulty.

A COMPARISON OF THE METHODS USED FOR INTERPRETING CREEP TEST DATA

By Joseph Marin[1]

Synopsis

There have been a number of methods developed for interpreting tension creep test data for the purpose of determining working stresses. All methods require extrapolation of test results beyond times covered by the tests. Various procedures have been used in making such extrapolations. It is the object of this paper to compare the working stress values as determined by the various methods. A comparison of results shows that there are appreciable differences in the working stresses as obtained by different methods of interpretation.

Introduction

In the design of members subjected to high temperatures the working stress for a given operating temperature is defined by a permissible plastic strain in a specified time. To determine such a stress, creep-time data are obtained and plotted as shown in Fig. 1. The curves shown are for tests made by Kanter[2] on 12 per cent chromium iron. To determine the working stress from such test results it is necessary to extrapolate the results to a time beyond that covered by the test. There are a number of methods used in doing this. A discussion of the various factors involved in the interpretation of creep test data and of the methods used is well summarized by Kanter.[3] It is of significance, however, to compare the working stress values as obtained by different methods of interpretation.

Methods of Interpretation

Method 1—McVetty's Method:

McVetty[4] assumes that the creep rate - time relation can be considered as

$$v - v_o = ce^{-\alpha t} \ldots\ldots\ldots (1)$$

where $v = \dfrac{d\epsilon_p}{dt}$ = the creep rate or slope of the creep-time curve,

t = the time, and

v_o, c and α = material constants for the particular stress and temperature.

The unit plastic or creep strain (ϵ_p) is by integration of Eq. 1:

$$\epsilon_p = \epsilon_o + v_o t - \frac{c}{\alpha} e^{-\alpha t} \ldots . (2)$$

In Eq. 2 as t becomes large, the creep ϵ_p approaches the value $\epsilon_p = \epsilon_o + v_o t$. That is, a constant creep rate is ap-

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[1] Assistant Professor of Engineering Materials, College of Engineering, Rutgers University, New Brunswick, N. J.

[2] J. J. Kanter and L. W. Spring, "Some Long-Time Tension Tests of Steels at Elevated Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 30, Part I, p. 110 (1930).

[3] J. J. Kanter, "Interpretation and Use of Creep Results," *Transactions*, Am. Soc. Metals, Vol. 24, No. 4, December, 1936, p. 870.

[4] P. G. McVetty, "Working Stresses for High-Temperature Service," *Mechanical Engineering*, Vol. 56, No. 3, March, 1934, p. 149.

proached asymptotically (Fig. 1). McVetty shows[4] that this function, with properly determined constants, represents quite accurately the actual creep-time curves. He assumes that for greater times than those covered by the tests, the plastic strain can be determined by the asymptote of Eq. 2, namely,

$$\epsilon_p = \epsilon_o + vt \text{..........} (3)$$

the constant ϵ_o representing the intercept on the creep axis (Fig. 1). Extrapolated creep-time relations as obtained by McVetty for various stresses are shown in Fig. 2 for a temperature of 800 F. To obtain a working stress value—a stress producing a specified creep in a selected time—a plot is first made between stress and percentage of creep using various times (Fig. 3). Such relations are obtained directly from Fig. 2. Thus for a creep of 1 per cent in 10 yr. the working stress for a temperature of 800 F. is the ordinate to the 10-yr. curve in Fig. 3. In the same way, stresses can be selected from Fig. 3 for other percentages of creep in 10 yr. For a temperature of 800 F. these stresses are then plotted as ordinates in Fig. 4. The stresses for other temperatures are also determined such that relations between stress and temperature for various creep values can be determined as shown in Fig. 4.

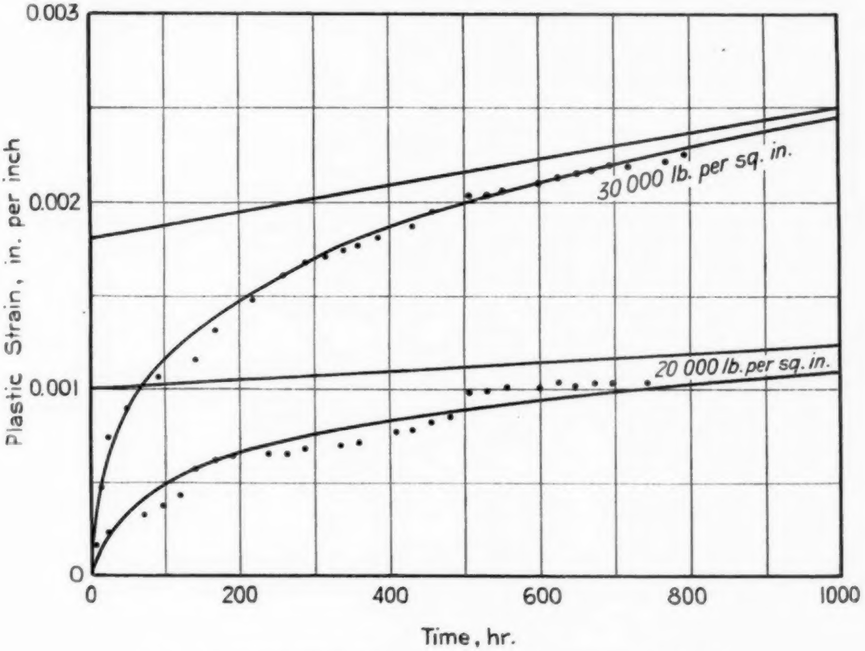

FIG. 1.—Creep-Time Relations for a Temperature of 800 F.

Method 2—Bailey's Method:

In a method used by Bailey[5] a series of creep-time curves are obtained for a

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[5] R. W. Bailey, "The Utilization of Creep Test Data in Engineering Design," Preprinted and read before the Institute of Mechanical Engineers (London), November, 1935.

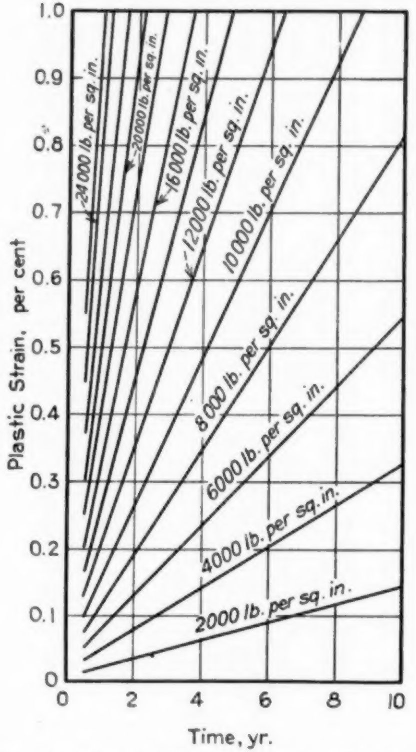

FIG. 2.—Extrapolated Creep-Time Relations for a Temperature of 800 F.

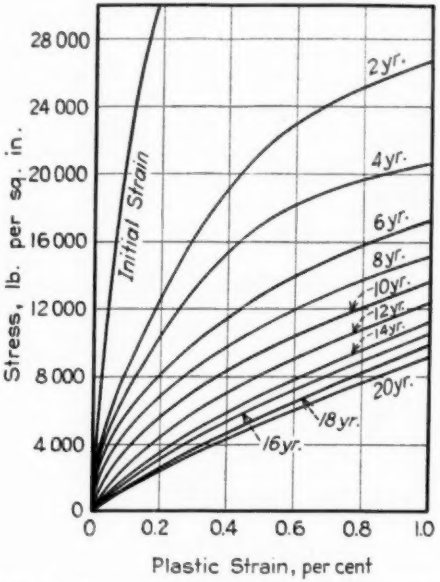

FIG. 3.—Stress-Creep Relations for a Temperature of 800 F.

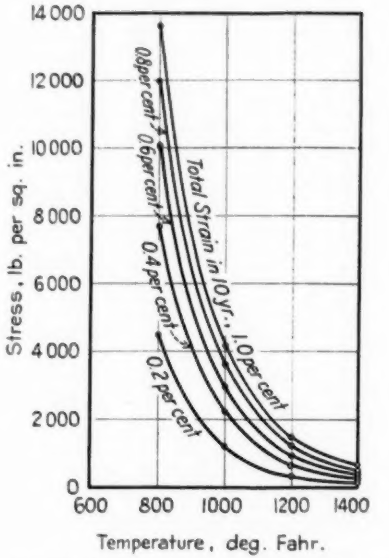

FIG. 4.—Working Stress - Temperature Relation (McVetty's Method).

material subjected to a given stress and tested at various temperatures. A plot of the results from each test is made as shown in Fig. 5, by plotting the logarithm of the time against the creep. The creep-time curves are then extrapolated as shown by the dotted lines to times beyond those covered by the tests.

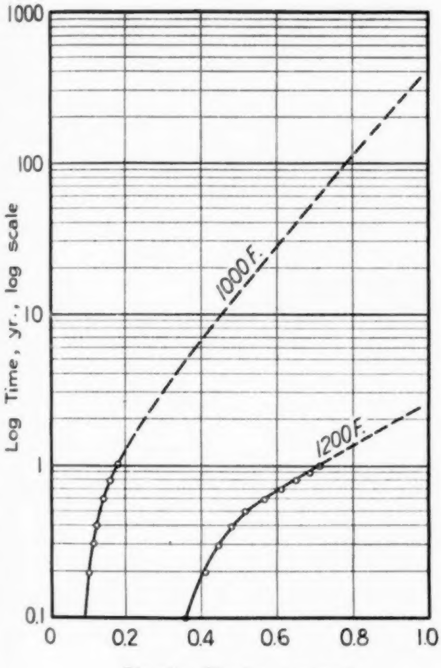

FIG. 5.—Log-Time-Creep Relations.
Stress, 4000 lb. per sq. in.

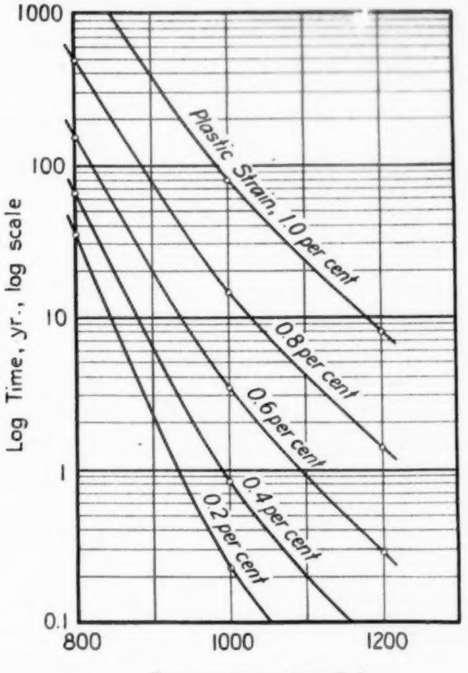

FIG. 6.—Log-Time-Temperature Relations.
Stress, 4000 lb. per sq. in.

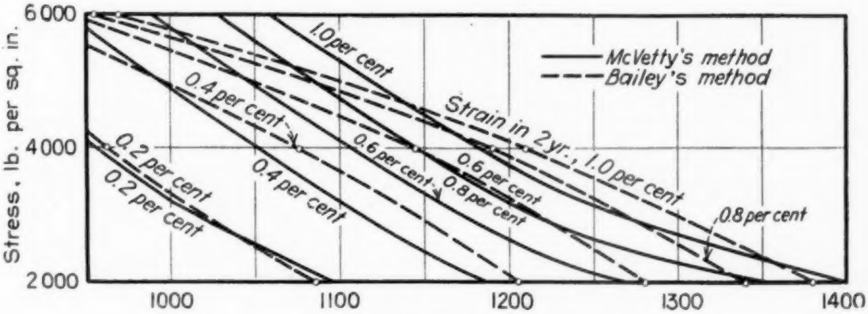

FIG. 7.—Comparison of McVetty and Bailey Methods.

From the relations between time and creep, the variation between temperature and time is determined for various percentages of creep (Fig. 6). The temperature to produce for the stress of 4000 lb. per sq. in., say an 0.8 per cent creep in 2 yr., can now be selected from Fig. 6. Temperatures corresponding to other creep values can be obtained in the same way. These values of temperature can be plotted as abscissa for the stress of 4000 lb. per sq. in. as represented in Fig. 7. Similar points are determined for other stress values such that relations between working stress and temperature can be plotted. These relations are shown by the dotted curves in Fig. 7.

Method 3—Log-Log Method:

A method which has been frequently used is to plot for a selected temperature the relation between the logarithm of stress and the logarithm of the minimum creep rate. A straight line is assumed to represent these test results and a prolongation of this line is assumed to give creep rates for stress values not covered by the tests.

Method 4—Log Method:

This method is similar to method 3. The stress in place of logarithm of stress, however, is plotted against the logarithm of the minimum creep rate. It is then assumed that the relation between stress and log creep rate is linear and extrapolation is made to determine the creep rates corresponding to stress values not covered by the tests.

Other methods of interpretation than the above are explained in papers by Kanter,[3] Soderberg[6] and Weaver.[7] For the purpose of comparison, only those stated above can be conveniently used.

COMPARISON OF METHODS

The author was unable to find complete creep test data for a material such that a comparison between the methods could be made. For this reason, methods 2, 3, and 4 were compared with McVetty's results as given in Figs. 1 to 4. That is, the creep-time relations shown in Fig. 2 were used as a basis of comparison. It is realized that this basis does not give accurate results. Such a procedure, however, shows in general the extent of divergence between the methods. For the Bailey method the stresses are shown in Fig. 7 for different creep rates. These results are based on an extrapolation of creep data from 1 to 2 yr. Figure 8 shows similar curves for extrapolations of data

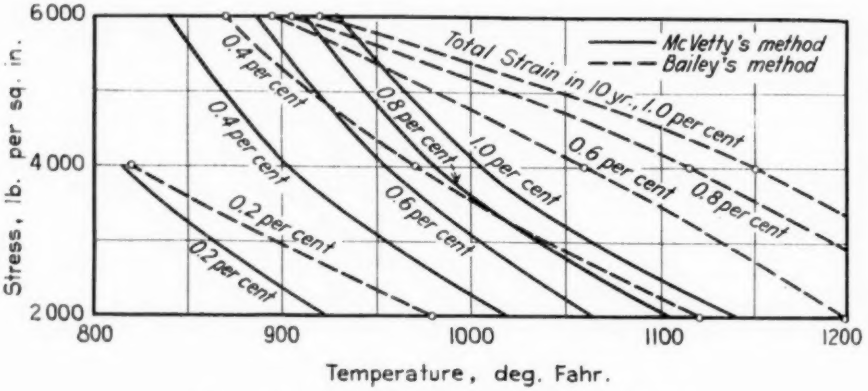

FIG. 8.—Comparison of McVetty and Bailey Methods.

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[6] C. R. Soderberg, "The Interpretation of Creep Tests for Machine Design," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 58, No. 8, November, 1936, p. 733.

[7] S. H. Weaver, "The Creep Curve and Stability of Steels at Constant Stress and Temperature," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 58, No. 8, November, 1936, p. 745.

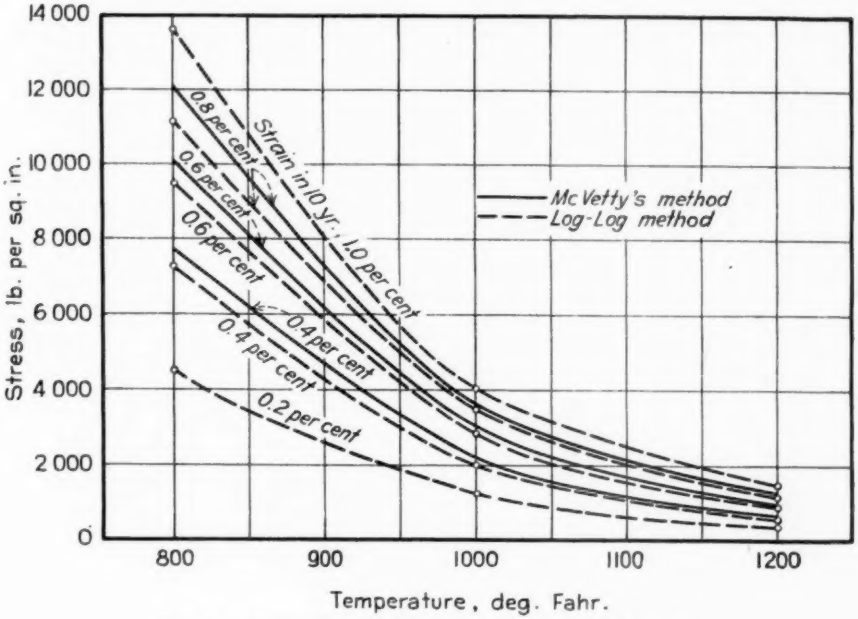

FIG. 9.—Comparison of McVetty and Log-Log Methods.

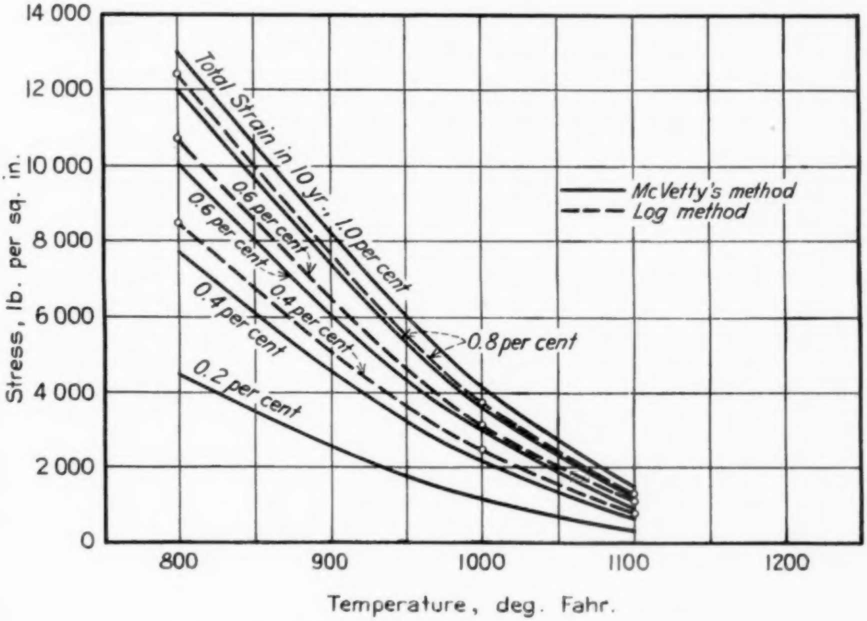

FIG. 10.—Comparison of McVetty and Log Methods.

from 1 to 10 yr. The maximum percentage divergence in stress for various temperatures as obtained from Figs. 7 and 8 are listed in Table I.

An estimate of the error in the log-log method is obtained by assuming a straight line in the log stress - log creep rate diagrams, between points representing 0.2 and 1.0 per cent in 10 yr. The stress-temperature relations in Fig. 9 are based on this procedure. Table II shows the maximum percentage divergences in stress for different temperatures.

The error in the log method is indicated using the above procedure for the log-log method. The stress-temperature relation based on this method is plotted in Fig. 10. The maximum per cent divergences in stress values are in Table II.

DISCUSSION AND CONCLUSION

The divergence in the stress values by Bailey's method indicates that considerable error may be introduced in extrapolation of test results to times much beyond those covered by the tests. By the log-log and log methods discrepancies in stress values indicate that these methods may give large errors. This is particularly true in view of the assumptions made in fitting the results to the stress-temperature relations for 0.2 and 1.0 per cent creep. It should be noted that the above results apply to a particular material, and that the creep-time curves were constructed on the basis of McVetty's assumed creep-time relations. The results indicate, however, that there may be considerable difference in the working stress value as obtained by different methods of interpretation.

TABLE I.—MAXIMUM DIVERGENCE IN STRESS FOR VARIOUS TEMPERATURES.

TEMPERATURE, DEG. FAHR.	DIVERGENCE IN STRESS, PER CENT	
	EXTRAPOLATION, 2 YR.	EXTRAPOLATION, 10 YR.
900	....	+28
1000	−18	+64
1100	+14	+119
1200	+22	

TABLE II.—MAXIMUM DIVERGENCE IN STRESS FOR VARIOUS TEMPERATURES.

TEMPERATURE, DEG. FAHR.	DIVERGENCE IN STRESS, PER CENT	
	LOG-LOG METHOD	LOG METHOD
800	−7	+8
1000	−10	+6
1200	−4	+20

DISCUSSION

MR. JOHN BOYD[1] (*presented in written form*).—It is interesting that the matter of the interpretation of creep data continues to attract attention in engineering circles. Such an interest is probably quite justified in view of the variance in the results between the different methods used at the present time.

Whenever an investigator attempts the problem of interpreting a set of creep test data, he immediately must give consideration to the manner in which the test data are plotted. Impressions gained from the appearance of such plotted data, together with preconceived thoughts on the matter and the desire to represent the findings simply, all influence the type of interpretations developed. The accompanying Figs. 1 to 4 show three of the more common sets of coordinates used in plotting relations between creep and time. These include: the regular plot; the semi-log plot; and the double log plot.

In the accompanying Fig. 1, which is the regular plot of percentage of creep *versus* time in hours, lines C and D represent uniform increases in strain of 0.1 and 0.01 per cent per year, respectively. These are ideal conditions, showing zero strain at zero time. Lines A and B also have slopes of 0.1 and 0.01 per cent per year, respectively, but show a positive strain of 0.05 per cent at zero time. This intercept of 0.05 per cent is arbitrary, but was chosen as a practical value since it was the same as the intercept of the asymptote to the creep curve, also shown in Fig. 1. This curve was taken from data which were distributed by Mr. S. H. Weaver of the General Electric Co. in conjunction with his paper on "The Creep Curve and Stability of Steels at Constant Stress and Temperature."[2] This particular test is plotted in Fig. 1 because it is typical of creep results and because, having run some 45,000 hr., it is the longest test yet recorded.

Figure 2 shows a semi-log plot of percentage of creep *versus* log time with this time scale extending to a service period of 20 yr. The lines A, B, C, and D of Fig. 1 are replotted in Fig. 2 and appear as exponential curves. The influence of considering or neglecting the intercept of the asymptote may be seen. Weaver's curve is also shown.

Figure 3 is a double log plot of the log of the percentage of creep *versus* the log of time. Again lines A, B, C and D, together with Weaver's curve, have been plotted. It should be noted that straight lines passing through the origin in the ordinary plot of Fig. 1 appear as straight lines inclined at 45 deg. in the double log plot of Fig. 3. Lines C and D may be associated with interpretations based on creep rate alone. Lines such as A and B are at first horizontal and later turn upward, finally approaching the straight lines C and D. The thing that is important to interpreters of creep test data is the location of the turning point along the time scale.

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[1] Research Engineer, Research Laboratories, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

[2] *Transactions*, Am. Soc. Mechanical Engrs., Vol. 58, No. 8, November, 1936, p. 745. The test shown is for a nickel-chromium-molybdenum steel at 842 F. and a stress of 25,000 lb. per sq. in.

For the practical case mentioned, it is seen that this point or region lies between 1000 and 100,000 hr. Weaver's curve[3] when shown on this plot is fairly straight for about the first 1000 hr. and then gradually turns upward.

Figure 4 has exactly the same coordinates as Fig. 3, but shows several different actual creep curves for various materials and temperatures. Curve 1 is a test by Sturm, Dumont and Howell on three-fourths hard 43S aluminum alloy at room temperature and 16,000 lb. per sq. in. stress.[4] Curve 2 is a test by McVetty on a carbon-molybdenum steel at 930 F. and 20,000 lb. per sq. in. stress. Curve 3 is a test by Cross and Lowther on 0.35 carbon steel at 850 F. and 7500 lb. per sq. in. stress.[4] Curve 4 is the test by Weaver already referred to. Curve 5 shows a creep test by the writer on 10 per cent prestretched copper at room temperature and a stress of 11,050 lb. per sq. in.

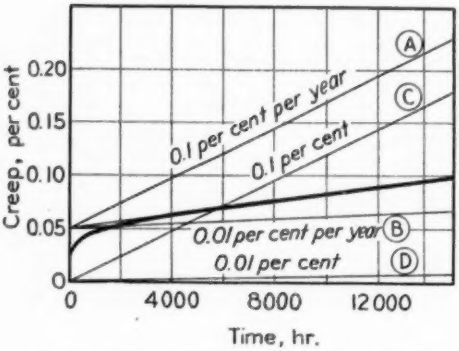

FIG. 1.—Creep-Time Curves—Regular Plot.

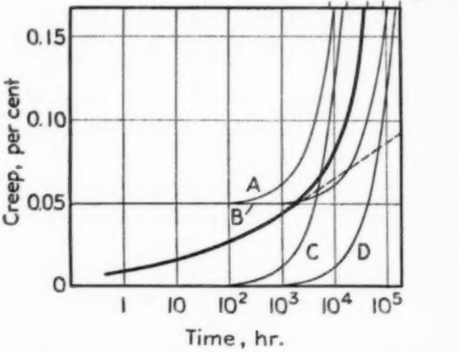

FIG. 2.—Creep-Time Curve—Semi-log Plot.

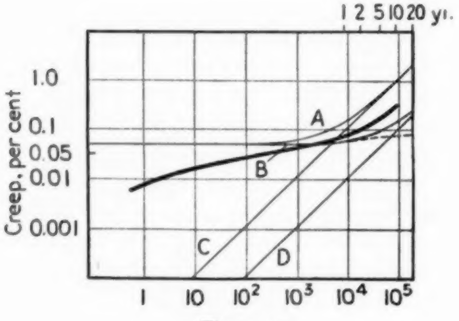

FIG. 3.—Creep-Time Curves—Double Log Plot.

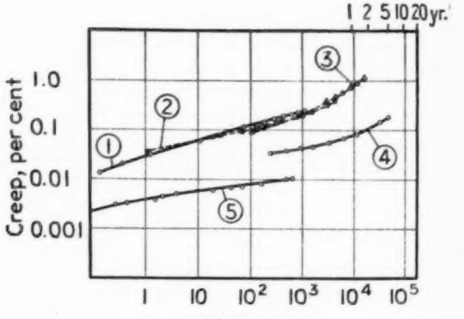

FIG. 4.—Creep-Time Curves—Double Log Plot.

Curves such as 1, 2, 3, 4 and 5 represent the extent of the data for which interpretation is to be made. Most tests do not run over 1000 to 2000 hr. If no test data beyond this time were available, one might easily conclude that a creep curve could be quite accurately represented by a power function expression of the time

$$\epsilon = at^n$$

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[3] Interpolated at the lower end by the writer.

[4] *Journal of Applied Mechanics*, Vol. 3, No. 2, June, 1936, p. A-62.

where ϵ = creep strain in per cent, t = time in hours, and a and n are constants. General expressions of this form have been suggested for creep. It must be pointed out, however, that tests that have run for longer periods of time all seem to indicate that a more or less uniform creep rate has been attained, and as a consequence, the creep curves gradually turn up, approaching a 45-deg. slope on double log paper.

Depending upon the use which is to be made of the creep data, the method of extrapolation may become very important. Drawing a straight line on one plot or another has been a rather popular manner of interpreting such data. However, if a straight line is drawn through the data of a 1000-hr. creep test and extrapolated, it will give an extension between five and ten times less than that which would be obtained by extending the creep curve to a 45-deg. slope or by assuming a constant strain rate.

In Fig. 2, a straight line has been drawn tangent to Weaver's curve at 1000 hr., after the manner of extrapolation sometimes used. This same line has been replotted in Fig. 3. When the creep in a period of 20 yr., as found by this line, is compared with the probable value which will be reached by Weaver's test, it is seen that the straight line on semi-log paper would show only about one-tenth the plastic strain given by the assumption of constant creep rate.

While it is not intended to convey the thought that all the various curves in Fig. 4 must gradually turn up at 45 deg. (or attain constant creep rates), it is again to be pointed out that several tests, together with the longest one on record, show this tendency. When comparing materials, it is probably not so important that long tests be run, and testing times of 1000 hr. and less may be sufficient. However, when the amount of allowable creep in a service time of, say 20 yr., is specified, the type of extrapolation used is quite important. In this latter case it may not be too safe to base general laws or extrapolation on tests of 1000 hr. duration unless the importance of the various factors involved is thoroughly understood. Eventually, long tests of many years duration may be available for checking the validity of various assumptions, but for practical purposes at the present time, interpretations such as those by McVetty and Weaver appear to fit the available test information very well.

MR. P. G. MCVETTY.[5]—The author has performed a valuable service in pointing out the effect of various methods of interpretation upon the conclusions drawn from creep data. It is evident that the proper use of such data for design purposes requires a clear understanding of several factors. These include methods of interpretation, details of the creep tests, the extent to which the test specimens are truly representative of the material being studied, and finally the metallurgical characteristics of the material. Without such intimate knowledge of the problem, it is doubtful whether data obtained in tests of a few months duration may be extrapolated safely to times representing the service life of the material.

Prior to 1933 there was a general tendency on the part of designers to consider creep rates only and to pay no attention to the fact that a tangent to the actual creep curve represented by the creep rate seldom, if ever, passes through the origin. The paper to which the author refers was written primarily to suggest a relatively simple method of considering the creep intercept at zero time.

Unfortunately, we do not know that the creep curve can be replaced by a

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[5] Mechanical Engineer, Research Laboratories, Westinghouse Electric and Manufacturing Co., East Pittsburgh, Pa.

straight line such as the final tangent or asymptote. Other curves have been suggested as a basis for straight-line extrapolation. By logarithmic or semi-logarithmic plotting, these methods assume a power function or exponential relation between creep and time.

Mr. Boyd has brought out a valuable point in connection with double logarithmic plotting. The fact that such curves cannot be extrapolated along a straight line to times representing the service life of the material if the original creep curve has an intercept, is a serious objection to this method of interpretation.

In general, the author has shown that the method which considers the creep curve intercept at zero time usually leads to more conservative estimates of working stress than methods based on creep rates only. With our present knowledge of the subject, it is impossible to claim great accuracy for any of the present methods of interpretation. It is expected that studies of the long-time tests now being conducted will help to clear up some of the existing uncertainties.

Mr. H. F. Moore.[6]—In the investigation of creep of lead, which has been in progress for several years at the University of Illinois, it has been found that lead at ordinary room temperature seems to behave very much like steel at elevated temperatures. In creep tests of lead and lead alloys it is nearly always possible to distinguish two stages, and, if the test is continued long enough, a third stage. In the first stage which may extend over one or two weeks for a tension specimen there occurs a rapid stretch which may be regarded as the gradual adjustment towards regularity of stress distribution in the specimen. In the second stage, the rate of stretch diminishes greatly, and approaches a straight-line relation between creep and time. In the third stage, the rate of creep again increases up to fracture. For lead and lead alloys, estimation of creep can, we believe, be satisfactorily made by the McVetty method, unless the creep gets into the third stage. This third stage seems to be the fracture stage, although for lead and lead alloys this stage may sometimes last over a long time —perhaps weeks or even months. In using creep data for design it would seem that, if the structural damage feared is the distortion due to non-fracturing creep, the McVetty method might be reliable, at least for lead and lead alloys. If the structural damage feared is fracture or cracking, tests extending into the third stage would seem desirable.

Mr. F. M. Howell.[7]—There is one point which we have observed in a number of creep tests and which may be of considerable importance. We have found that when the curve obtained by plotting the logarithm of creep as ordinates and the elapsed time as abscissas departs from a straight line there is also a slight change in the cross-sectional dimensions of the specimen, thus indicating, as Mr. Moore has brought out, that the departure from the straight line may show the beginning of structural damage.

Mr. Joseph Marin.[8]—The information given by Messrs. Boyd, McVetty, Moore and Howell on the interpreting of creep test data is important in showing the discrepancies encountered in using various methods of interpretation. A method of interpretation not included in the paper by the writer is one developed by Soderberg.[9] A consideration of this method for several tests shows that it gives a closer agreement with test results than the interpretations considered above.

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[7] Engineer of Tests, Aluminum Research Laboratories, Aluminum Company of America, New Kensington, Pa.

[8] Asistant Professor of Engineering Materials, College of Engineering, Rutgers University, New Brunswick, N. J.

[9] C. R. Soderberg, "The Interpretation of Creep Tests for Machine Design," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 58, No. 8, November, 1936, p. 733.

THE CELITE TYPE HIGH-TEMPERATURE THERMAL CONDUCTIVITY APPARATUS

By C. E. Weinland[1]

Synopsis

This paper will bring up to date the description of this type of apparatus, four units of which are now in daily use for testing thermal insulation at temperatures up to 1600 F. The present method of constructing heater plates is described, as is also the assembly of the apparatus with metallic isothermal plates and heaters for the "cold" faces of the test samples, all of which are modifications introduced since the previous description was published. A simplified arrangement of the thermocouples is now used, and for electrical power measurement a wattmeter circuit is described which provides wattage readings over an extended range on a large number of power circuits, with the elimination of certain troublesome circuit errors and with the minimum of instrumental equipment. The method of conducting the test makes possible results in a form approximating the mathematical concept of conductivity, with attendant advantages in the use of the results in heat-loss calculations. Typical test results and calculations are shown.

The Celite type apparatus for measuring thermal conductivity at high temperatures, originally built in the Celite Co. Research Laboratory at Lompoc, Calif., has been described by Calvert and Caldwell[2] and by Hartmann, Westmont and Weinland.[3] Since the publication of the latter paper a number of important changes have been made in apparatus design and mode of operation, by the staff of the Johns-Manville Research Laboratories. It is the purpose of this paper to bring up to date the description of apparatus and method of testing.

Main Heater Plate:

This plate is now wound upon a spirally grooved Alundum plate supplied by the Norton Co., Worcester, Mass.[4] The use of platinum heating elements has been discontinued, for the heater plate temperature is limited to 1600 F. as will be mentioned later, and at this temperature or lower the life of Nichrome V No. 22 B. & S. gage has been found comparable to that of the best platinum heater alloy. In winding the wire in grooves, the use of frequent spots of de Khotinsky cement is required, due to the stiffness of the wire, in order to hold it in place until the cover plate (a $1\frac{1}{4}$-in. thick alundum filter plate, RA98) has been cemented in

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[1] Physicist, Johns Manville Corp., Manville, N. J.

[2] R. Calvert and L. Caldwell, "Heat Loss from Furnace Walls," *Industrial and Engineering Chemistry*, Vol. 16, No. 5, p. 483 (1924).

[3] M. L. Hartmann, O. B. Westmont and C. E. Weinland, "Methods of Measuring the Thermal Conductivity of Insulating and Refractory Materials," *Proceedings*, Am. Soc. Testing Mats., Vol. 28, Part II, p. 820 (1928).

[4] Blueprint No. 21025, Norton Co., Worcester, Mass.

place. The method of winding center and edge coils, and the approximate location of the potential taps are shown in Fig. 1.

Figure 1 also shows the location of the differential couple used to detect temperature balance between center and edge of this plate. Since the 1928 paper[3] the wires have been so disposed as to shorten them materially, and platinum and platinum-rhodium have been interchanged in position, so that the external leads are of the alloy wire which is less subject to breakage from continued handling.

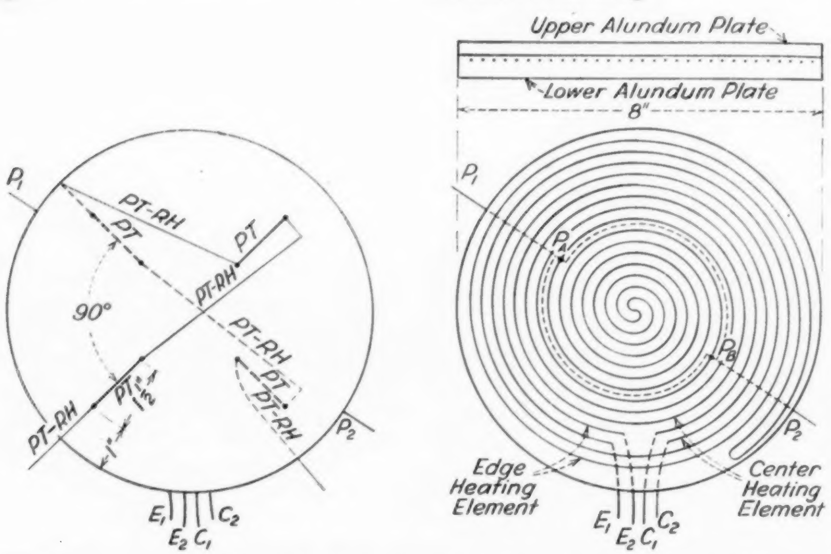

FIG. 1.—Arrangement of Differential Thermocouple and Heater Winding in Main Heater Plate. Dotted lines enclose the "area of power measurement."

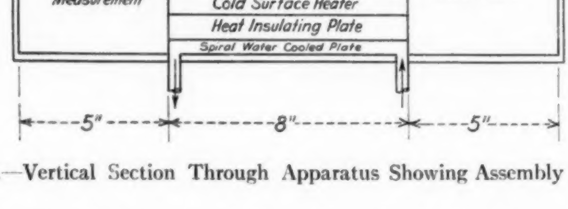

FIG. 2.—Vertical Section Through Apparatus Showing Assembly of Parts.

Assembly of Parts:

The arrangement of the parts of the apparatus is shown in elevation in Fig. 2, the entire assembly being surrounded by Sil-O-Cel insulating powder contained in a cylindrical shell as shown in Fig. 3.

The heat developed in the "Area of Power Measurement" (Fig. 2) divides, part flowing upward through one sample, the remainder flowing downward through the other sample. The measured heat is forced to flow in parallel lines, perpendicularly to the faces of the samples (assuming the samples to be uniform and isotropic) by the fact that the faces of the heater plate are substantially isothermal, and may be so maintained by observation of the differential thermocouple and manipulation of the edge coil current control. This design has been criticized[5] as lacking metal faces for the heater plate and complete division between center and edge structures, but experimental verification of the necessity of these features is at present lacking.

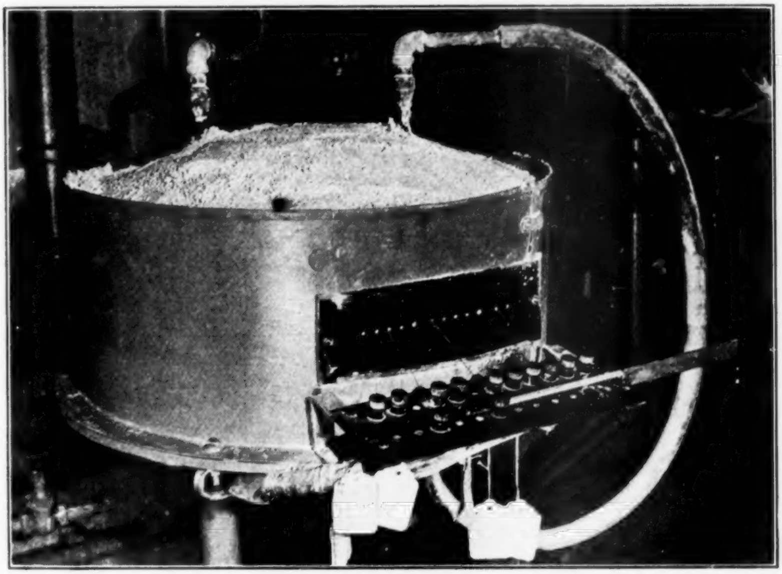

FIG. 3.—Exterior View of Assembled Apparatus.

The winding of the section of the main heater between the "Area of Power Measurement" and the edge coil is a continuation of the former winding, and acts as a second guard, to improve assurance of attainment of the isothermal condition over the area of power measurement.

In contact with the "cold" face of each sample there is a $\frac{1}{4}$ in. thick plate of pure nickel, of diameter equal to the main heater and samples (8 in.). These plates, by virtue of their high thermal conductivity, serve to establish isothermal surfaces on the "cold" faces of the samples. They are separated from the samples by sheets of asbestos paper $\frac{1}{32}$ in. thick, which are necessary to prevent short-circuiting the thermocouples in contact with the "cold" faces of the samples.

In order that the "cold" faces of the samples may be maintained at temperatures of the order of 150 deg. Fahr. lower than that of the hot faces (that is, at temperatures up to 1450 F.) there is in contact with each isothermal plate a "cold-surface heater." These heaters

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[5] P. Nicholls, "Determination of the Thermal Conductivity of Refractories," *Bulletin*, Am. Ceramic Soc., Vol. 15 No. 2, p. 37 (1937).

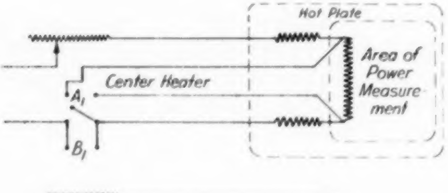

FIG. 4.—Schematic Wiring Diagrams of Heater Windings.
A Wattmeter potential terminals.
B Wattmeter current terminals.

are wound upon the same type of grooved Alundum plate and with the same heater wire and cover plate as the main heater, but without potential leads from the central section, and with the main winding continued to the edge of the plate in place of a separate edge winding. Each is supplied from its own current control, so that the "cold" faces of the two samples may be maintained at substantially the same temperature.

Insulating plates are placed in contact with each cold surface heater, and spiral tube water-cooled plates in contact with each insulator, in order that the entire arrangement may be as nearly independent of room temperature fluctuations as possible.

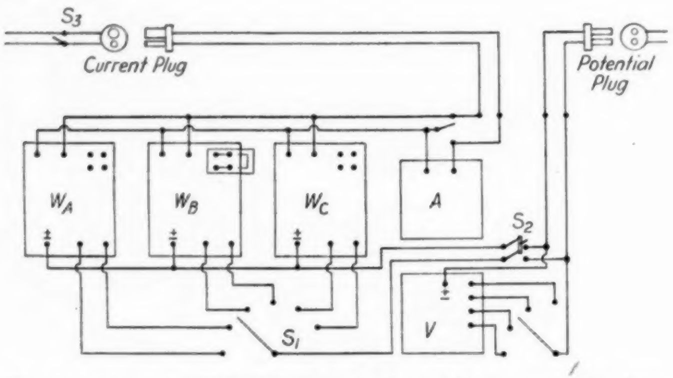

FIG. 5.—Schematic Wiring Diagram of Power Measurement Carriage.

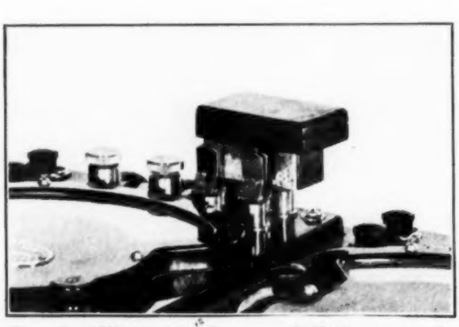

FIG. 6.—Wattmeter Current Coil Connector in "Coils in Parallel" Position.

Electrical Circuits:

The power supply for the entire laboratory, including at present four units of Celite type apparatus, five units of "Bureau of Standards" type low-temperature apparatus, two units of Globar type high-temperature apparatus and fifteen units of pipe covering conductivity apparatus, is 112-v., 60-cycle a.c. derived from a generator with Thyratron voltage control of the type described by the author[6] but since modified for full-wave Thyratron rectification. A separate auto transformer for each unit of apparatus supplies power to the switchboard in 7-v. steps, and in each line there is a constantan wire rheostat for fine control of current and power. The electrical circuits for center and edge windings of the main heater, and for one cold-surface heater are shown in Fig. 4.

Power-Measuring Circuit:

In order to avoid costly duplication of wattmeters, and at the same time to have available for every line on the switchboard a wattmeter circuit of good accuracy and wide range a measuring unit has been in use since 1930, and not heretofore described. Figure 5 shows the electrical circuit. The unit consists of a movable carriage on which are mounted three Model 310 Weston wattmeters and a Model 425 Weston high-resistance thermocouple type voltmeter. Wattmeter *A* has three full-scale ranges obtained by proper selection of potential and current coils, giving full-scale deflection on either 4, 8 or 16 watts. Meter *B* has full-scale ranges of 30, 60 and 120 watts while meter *C* has ranges of 250, 500 and 1000 watts. By proper selection of meter and range it is possible to read any wattage value between 2 and 1000 watts at half scale or more on a meter with a nominal accuracy of one-quarter of 1 per cent of full-scale value. The selection of one of two potential coils in either of the three wattmeters is accomplished by the 6-point rotary switch S_1. The current circuits of all three meters are connected in parallel, but all are open circuited until appropriate binding posts on the faces of the meter are connected. Adjustable links are supplied by the manufacturer for connecting these posts, as is known by any user of this type of instrument, but in the present application the operation has been made much more rapid by the use of a special blade-type connecting device shown in Fig. 6 in the "coils in parallel" position, and shown diagrammatically on Fig. 5.

Since the wattmeter is in the circuit of any one heater only while the wattage measurement is being made, a correction must be applied to take account of the disturbance in the circuit caused by insertion of the wattmeter and associated lines. This is accomplished by reading the voltage (E_1) on the potential line before connecting in the wattmeter potential coil (with switch S_2 of Fig. 5 open) and also before shunting the current through the wattmeter current circuit (with switch S_3 of Fig. 5 closed). The voltmeter has four ranges, 7.5, 30, 75 and 150 v., and has a resistance of 125 ohms per volt, so the power drain of the voltmeter is negligible. A second voltage reading, E_2, is taken on the potential line with both wattmeter current and potential coils in circuit, and the wattmeter reading, W_I, multiplied by $\frac{E_1^2}{E_2^2}$, is taken as the true wattmeter reading for the undisturbed line (assuming the resistance of the line and heater to remain constant during the reading). This wattage value must be further corrected, however, for the current drain of the wattmeter potential coil must necessarily come through the wattmeter current coil, so a wattage equal to $\frac{E_2^2}{R_P}$, where R_P is the resistance of the particular potential coil in use, is subtracted from the indicated reading to give the true wattage. Thus the true watts $W_T = W_I \frac{E_1^2}{E_2^2} - \frac{E_2^2}{R_P}$. The two corrections tend to be in opposite directions, and

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[6] C. E. Weinland, "The Thyratron Voltage Regulator for an Alternator," *Review of Scientific Instruments*, Vol. 3, No. 1, p. 9 (1932).

may cancel or be of negligible magnitude at large power values with small currents, but tend to increase in importance at lower wattage values.

Thermocouples:

In contact with each face of each of the two specimens there are placed two platinum (90 per cent platinum, 10 per cent rhodium) thermocouples No. 28 B. & S. gage. Sheets of asbestos paper on both faces are sufficiently soft that the thermocouples will imbed themselves in the paper. The ends of the couples are brought out through the outer shell of the apparatus where they are connected by binding posts (visible in Fig. 3) to copper leads leading to a plug and jack board (not shown) at the type *k* potentiometer position. The temperature of the couple-to-copper junction is taken by an ordinary mercury thermometer.

These couples are frequently checked in calibration against a standard couple certified by the Bureau of Standards, and the portions of the couples in contact with the samples are discarded and replaced by new wire when found to be appreciably out of calibration, if they cannot be returned to good condition by annealing.

Preparation of Samples:

The standard sample size is a disk $1\frac{1}{8}$ in. in thickness and 8 in. in diameter, of which two as nearly identical as possible are required. Faces are kept plane and parallel to within ±0.005 in. Samples made up from bricks are cut so that each is made of three pieces, one $4\frac{1}{4}$ to $4\frac{1}{2}$ in. wide, constituting the central section, the other two being segments to complete the circular shape, thus keeping joints outside the 4-in. area of power measurement.

Conduct of Test:

In conductivity testing of this type, it is desirable to keep the temperature drop through the sample to a minimum for two reasons. In the first place, if the entire volume of the sample is maintained at a temperature approximating that of the hot face, the effect of any chemical or physical changes affecting the conductivity will be shown more clearly by the results than would be the case if the temperature drop through the sample were of a magnitude of hundreds of degrees. In the second place, the use of the results in the calculation of the heat loss through the material when used in some projected design of equipment is often facilitated when the conductivity data is in the form of a "true" conductivity,[7] that is, in the form suggested by the basic Fourier equation $dq = k \frac{dt}{dx}$ where dq is the time rate of heat flow through a unit cube of the material under a temperature gradient $\frac{dt}{dx}$, and the conductivity k is understood to be a function of temperature.

It is believed that in practice the "true" conductivity function may be sufficiently well approximated by maintaining a temperature difference of the order of 150 F. between hot and cold faces of the sample. Smaller temperature differences would unduly increase the relative error arising from uncertainties in temperature measurements, unless special means were employed to reduce them.

Consequently the operation of the apparatus consists in applying an amount of power to the main heater calculated to give the temperature drop desired, and amounts of power to each

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[7] C. E. Weinland, "A Graphical Method of Calculating Heat Loss Through Furnace Walls," *Journal*, Am. Ceramic Soc., Vol. 19, No. 3, p. 74 (1936).

cold surface heater found by experience to give the desired cold surface temperature, plus an amount of power in the edge heater as required to give zero potential on the differential couple of the main heater. When temperature balance and temperature constancy have been attained (generally 24 hr. or longer) a thermal conductivity value is calculated and associated with the mean temperature of the sample. Five such points at as many values of mean temperature will generally give a good picture of the behavior of a commercial insulating material. Points should be taken, except in very special cases, with successively increasing temperatures, for the irreversible thermal changes in the sample may be relatively large.

The maximum temperature practically attainable with apparatus of this design is 1600 F., for above this point alundum has been found to become sufficiently conductive of electricity to cause power losses[3] and to cause current leakage from heater circuits to thermocouples. This effect has been repeatedly observed with a number of different main heaters.

TABLE I.—TYPICAL RESULTS OF TEST.

Johns-Manville Superex Blocks.

Main heater power setting 10.0 watts. Lower cold surface power setting 120 watts. Upper cold surface power setting 120 watts.

Date and Time	Cold Junction Temperature, deg. Fahr.	Thermocouple Readings, mv.								Voltmeter Readings		Wattmeter Readings		Wattmeter Readings		Differential Center to Edge, mv.
		Lower Cold Surface		Upper Cold Surface		Lower Hot Surface		Upper Hot Surface								
		1	2	3	4	5	6	7	8	E_1	E_2	W_1	Edge	Cold Surface 1	Cold Surface 2	
10/15/36																
8:30	81	5.10	5.14	5.13	5.16	6.75	6.74	6.73	6.72	10.0	9.8	10.0	51.8	120.2	120.0	0.01+
9:30	81	5.10	5.14	5.13	5.16	6.75	6.73	6.73	6.72	10.0	9.8	10.0	52.0	120.0	120.0	0
10:30	82	5.09	5.14	5.12	5.15	6.77	6.74	6.74	6.73	10.0	9.8	10.0	52.0	120.5	120.0	0
11:30	83	5.08	5.13	5.11	5.16	6.78	6.76	6.74	6.74	10.0	9.8	9.9	52.0	120.0	120.0	0
12:30	84	5.08	5.13	5.12	5.16	6.79	6.76	6.75	6.74	10.0	9.8	10.0	52.0	120.0	120.0	0.01−
1:30[a]	83	5.07	5.12	5.12	5.15	6.79	6.76	6.75	6.74	10.0	9.9	10.0	52.1	120.3	120.1	0
2:30[a]	82	5.06	5.12	5.11	5.15	6.77	6.75	6.74	6.73	10.0	9.8	10.0	52.0	120.0	120.0	0
3:30[a]	82	5.07	5.13	5.11	5.15	6.77	6.75	6.73	6.73	10.1	9.8	10.0	52.0	120.0	120.0	0
4:30[a]	81	5.08	5.14	5.11	5.14	6.76	6.75	6.73	6.73	10.0	9.8	10.0	52.0	120.0	120.0	0
Averages	82	5.07	5.13	5.11	5.15	6.77	6.75	6.74	6.73	10.0	9.8	10.0				

[a] Last four readings only, included in average.

Accuracy of Results:

It can be broadly stated that in the course of day-to-day work with four similar units of apparatus of this design, the self-consistency of the results may be expected to be within ±5 per cent, both in repeat testing and in parallel testing in different units. The question of absolute accuracy, however, cannot be adequately discussed here. In the first place the practical difficulties attendant upon its determination have prevented extended work along this line. In the second place, results with this type of apparatus have been compared with those secured in several other laboratories by competent investigators all using samples from the same lot of uniform material, with widely divergent results. As a result of this comparison the Bureau of Standards in cooperation with the Subcommittee on Heat Transfer of the Society's Committee C-8 on Refractories, have undertaken a fundamental study of the causes of error in high-temperature conductivity testing.

Observations and Calculations:

To show a sample set of test observations and results the data in Tables I and II are given, the material tested being Johns-Manville Superex.

From these data the conductivity is calculated as shown in Table III.

Mean data from four other test points on the same sample gave conductivities shown in Table II.

TABLE II.—CONDUCTIVITY-MEAN-TEMPERATURE DATA.

MEAN TEMPERATURE, DEG. FAHR.	CONDUCTIVITY
427	0.641
733	0.685
1095	0.770
1265	0.830
1474	0.892

TABLE III.—CALCULATED CONDUCTIVITY.

Millivolts	5.07	5.13	5.11	5.15	6.77	6.75	6.74	6.73
Cold junction correction	+0.16	0.16	0.16	0.16	0.16	0.16	0.16	0.16
	5.23	5.29	5.27	5.31	6.93	6.91	6.90	6.89
Calibration correction	+0.02	−0.02	−0.01	−0.01	+0.03	+0.03	+0.04	+0.03
Millivolts corrected	5.25	5.27	5.26	5.30	6.96	6.94	6.94	6.92
deg. Fahr.	1117	1120	1119	1126	1410	1408	1408	1404

Average cold surface temperature = 1121 F. Average hot surface temperature 1408 F.

$$\text{Temperature difference} = 1408 - 1121 = 287 \text{ F.}$$

$$\text{Mean temperature} = \frac{1408 + 1121}{2} = 1265 \text{ F.}$$

Area of power measurement $= A = 11.848$ sq. in.
Heat loss through measured area $= Q = 3.415 \times W$ B.t.u. per hour where W = watts
Thickness of sample $= l = 1.117$ in.
Temperature difference $= T_2 - T_1 = 287$ F.

$$k = \frac{Ql}{2A(T_2 - T_1)} = \frac{3.415\ Wl}{2A(T_2 - T_1)} = \frac{K\ Wl}{(T_2 - T_1)} \text{ where } K = \frac{3.415 \times 144}{2 \times 11.848}$$

$$K = 20.75$$

$$k = \frac{20.75\ Wl}{T_2 - T_1}$$

W_I	E_1	E_2	$W\frac{(E_1)^2}{(E_2)^2}$	$\frac{E_2^2}{R_P}$	$\frac{W_T}{10.3}$
10.0	10.1	9.6	10.6	0.3	10.3

$$\text{Finally } k = \frac{20.75 \times 10.3 \times 1.117}{287} = 0.830 \text{ B.t.u. in. per hr. per sq. ft. per deg. Fahr.}$$

TRICALCIUM ALUMINATE AND THE MICROSTRUCTURE OF PORTLAND CEMENT CLINKER

By Levi S. Brown[1]

Synopsis

Recognition of C_3A in portland cement clinker by microscopic examination has been found exceedingly difficult. To determine its mode of occurrence, that is, its relation to the structure of clinker, about 150 experimental burns were made ranging from pure C_3A through high-alumina silicate mixes into the field of portland cement compositions. The charges were examined in thin sections, especially prepared to yield optimum clarity.

It is found that C_3A crystallizes in large units, enclosing C_3S, and CaO when present, but with a tendency to exclude C_4AF. In this habit C_3A becomes a matrix or interstitial material which, because of its close similarity in optical properties to those of C_3S, its presence usually in minor amounts, and general lack of crystal outlines in silicate mixes, is quite difficult to distinguish. Its presence is demonstrated indirectly through its control over the orientation of C_4AF crystallites, which show a common mass or field birefringence over large, roughly equidimensional areas. The common lack of such criteria in commercial clinker indicates that crystalline C_3A is not present in the amounts generally calculated.

Petrographers studying portland-cement clinker long have experienced great difficulty in recognizing crystalline C_3A. The investigation reported herein was undertaken to study its mode of occurrence, using thin sections to facilitate correlation of structures developed with relationships indicated in phase-equilibrium diagrams. The thin sections were mounted in Hyrax (**1**)[2] to minimize relief, and the specimens were ground in oil to eliminate hydration products.

Experimental clinkers were prepared to trace the behavior of C_3A from the binary system $CaO\text{-}C_5A_3$ through the ternary system $CaO\text{-}C_5A_3\text{-}C_2S$ and the quaternary system $CaO\text{-}C_5A_3\text{-}C_2S\text{-}C_4AF$ into clinkers of commercial manufacture. About 150 experimental burns were made, using a small platinum-wound electric muffle.

The Binary System $CaO\text{-}C_5A_3$

Specimen 1.—A finely ground mix of pure C_3A composition, heated 2 hr. at 1425 C., sinters into a hard tough clinker. The thin section reveals a dense mass of small C_3A crystals, hexagonal to rectangular in outline and averaging 10 μ in diameter, set in a formless isotropic matrix of lower index. Occasional grains of free CaO are noted.

Specimen 2.—Reheating specimen No. 1 at 1520 C. for 30 min. caused a partial recrystallization of the C_3A into large units, from 0.5 to 2.0 mm. or more on a side, representing combinations of

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[1] Research Petrographer, Lone Star Cement Corp., Hudson, N. Y.

[2] The boldface numbers in parentheses refer to the reports and papers given in the list of references appended to this paper, see p. 305.

the cube and octahedron. There is some reduction in the amount of free CaO. The finely and coarsely crystalline products are shown in Plate I. Well rounded vacuoles represent partially the voids in the original powder, the balance being taken up as shrinkage in sintering.

Specimen 3.—When the pure C_3A mix is heated to the dissociation temperature, 1535 C., substantial amounts of free CaO, in the form of clear yellowish rounded grains, and liquid are formed. On cooling, C_3A crystallizes very rapidly from the liquid in the form of cubes, usually with the octahedron, and frequently the rhombic dodecahedron also, well developed. In this crystallization the grains of free CaO serve as crystallization nuclei, as might be expected from the fact that C_3A is a higher lime compound than the liquid. These relations are shown in Plate II. This observation is of distinct practical importance. It is evident that the grains of CaO are thus isolated from contact with the liquid, and the attainment of complete equilibrium crystallization would require an exceedingly lengthy and careful annealing, as pointed out many years ago by Shepherd, Rankin, and Wright (3).

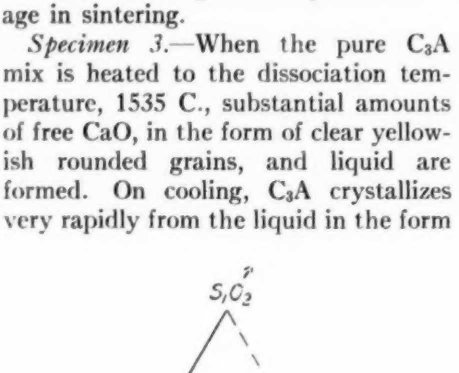


FIG. 1.—Portion of $CaO-Al_2O_3-SiO_2$ Diagram, Showing Compositions Described.
(After Rankin and Wright.)

Specimen 4.—A charge of CaO 52.88 per cent, Al_2O_3 47.12 per cent, (C_3A 35 per cent, C_5A_3 65 per cent) was prepared for the purpose of observing the behavior of C_3A and the C_3A-C_5A_3 eutectic, on the C_3A side of the eutectic ratio. The charge was heated to 1500 C., considerably above the eutectic temperature, 1395 C., to secure complete fusion. From this temperature the charge was slowly cooled through a period of 55 min. to 1390 C.

The thin section of this charge reveals the excess of C_3A as large phenocrysts, usually with regular inclusions of C_5A_3, as illustrated in Plate III. The phenocrysts are combinations of the cube and octahedron. The crystal structure of the enclosing eutectic (Plate IV) is that of blade-like spears of C_3A, representing a succession of partly intergrown cubes, and small skeletal crystals, set in a formless mass of lower refractive index.

CaO-Al_2O_3-SiO_2 RELATIONS

A portion of the original ternary diagram (2) is shown in Fig. 1. To study the behavior of the lime aluminates in the presence of silica, burns were made of compositions represented by points along the lines C_3A-C_3S, C_3A-C_2S, at 10 per cent intervals, with quite a number of compositions between the two lines. In some cases very peculiar structures were observed, structures not clearly understood. In general, however, the coarsely crystalline habit of C_3A was again in evidence, together with other interesting structures substantiating the course of crystallization as indicated by the diagram.

At the outset, attention should be directed to the two invariant points, 17 and 16, with temperatures of 1470 C. and 1455 C., respectively. In any mix on or to the left (the high lime side) of the line C_3A-C_3S no stable liquid can be developed below a temperature of 1470 C. Similarly, a stable liquid will not appear below 1455 C. in any mix along the line C_3A-C_2S or between that and the line C_3A-C_3S (not including this latter line). On cooling any melt of composition on or to the left of the dotted lines 17-C_3S, 17-C_3A, the liquid composition will pass through point 17. If the mix is to the left of but not on these lines, the liquid will enclose, at point 17, some free solid $CaO \cdot C_3A$ begins to crystallize as point 17 is reached. With equilibrium maintained, the liquid composition will remain at point 17 until all of the excess lime, or all of the liquid, has disappeared. The melt of any mix to the right of, but not on, the line C_3A-C_3S, up to and including the line C_3A-C_2S will eventually reach point 16, where C_2S begins to crystallize. The liquid composition here is deficient in lime, and the deficiency must be made up by dissociation of previously formed C_3S. The dissociation does not necessarily indicate the resolution of C_3S as indicated by Lea and Parker (4). The dissociation products are CaO and C_2S, but the liquid is already saturated with C_2S. Thus the C_2S formed appears as an alteration border around the C_3S crystals, as will be illustrated presently, and in some cases completely altered pseudomorphs have been observed.

Five specimens representative of different mixes and thermal treatment are presented, as itemized below:

1. A mix on the C_3A-C_3S line above the point *a*, that is, where it is crossed by a line from CaO to point 17, burned at a temperature above 1470 C. Above this point, C_3S will separate from the melt prior to C_3A.
2. A mix on the C_3A-C_3S line below point *a*, such that C_3A separates prior to C_3S, burned at a temperature above 1470 C.
3. A mix on the C_3A-C_2S line where C_2S separates prior to C_3A, burned above 1470 C.
4. A mix on the C_3A-C_2S line where C_3A separates prior to C_2S, burned above 1470 C.
5. A siliceous mix moderately high in alumina burned at a temperature slightly below that of any stable liquid formation.

Specimen 1.—Mix: C_3A, 40 per cent, C_3S, 60 per cent. Heat treatment: 2 hr. at 1490 to 1500 C.; cooled slowly through 30 min. to 1450 C. Good shrinkage, but very slight slump.

OBSERVATIONS: (*a*) With plane polarized light: A seemingly formless matrix, with index corresponding for the most part to C_3A, enclosing quantities of small C_3S crystals, rather well formed and uniform in size; average 25 to 30 μ across. Free CaO grains rather numerous, but not uniformly distributed; occur in segregated bunches (Plate V*a*). (*b*) With crossed nicols: Plate V*b* shows the same field as Plate V*a*. There are observed irregular but roughly equidimensional areas, dark and nearly isotropic, separated by paths containing an abundance of brightly birefringent C_2S flakes. The isotropic areas enclose the segregations of free CaO grains. On closer inspection the bright C_2S flakes are found to be alteration borders on C_3S crystals. On the other hand, C_3S crystals are just as numerous within the isotropic areas as elsewhere, but they show no trace of alteration borders.

INTERPRETATION: The charge was heated above 1470 C.; hence, in composition the liquid formed followed up the C_3S-CaO boundary for a short distance. On cooling, the liquid returned along this boundary, arriving at point 17 enclosing an abundance of C_3S crystals and some free CaO grains. At point 17, C_3A began to crystallize, with the free lime grains as nuclei. The isotropic areas represent the initial crystallization of C_3A; its habit of forming large crystals is again illustrated, now reaching out and crystallizing around the C_3S crystals. With the CaO removed from contact with the liquid, the latter moved on in composition to point 16, as cooling progressed. Here C_2S began to form in the liquid areas remaining between the C_3A crystals, giving rise to the alteration on the enclosed C_3S crystals, as previously described. Alteration borders obviously could not form on the C_3S crystals enclosed within the already solid crystalline C_3A.

Crystal boundaries of the large isotropic C_3A areas are generally not defined for the reasons that (1) the crystals merge into one another and, lacking birefringence, there is no way to tell where one leaves off and the other begins, and (2) crystallization of C_3A is continuous from point 17 through point 16 to point 15. Occasionally, straight sharp borders of C_3A can be seen against the lower index matrix; when so observed the corners are sharp right angles, indicating the cube as the only form developed.

The structures described in the preceding paragraphs are typical of all mixes on the C_3A-C_3S line above point *a*, that is, the point above which C_3S crystallizes prior to C_3A, when heated above 1470 C. However, when C_3A is present in the mix in proportions of 10 per cent or less it becomes exceedingly difficult to distinguish any trace of matrix material. The whole structure is very lace-like and porous. Burning is very difficult, shrinkage is slight, and the only minerals that can be distinguished are C_3S in irregularly-shaped flakes and large flakes of residual C_2S, with substantial amounts of residual CaO, roughly recrystallized.

Specimen 2.—Mix: C_3A, 80 per cent, C_3S, 20 per cent. Heat treatment: 1 hr. at approximately 1500 C. Charge melted enough to slump completely. Annealed 40 min. from 1500 C. to 1450 C.

Observations: (*a*) With plane polarized light. Large cubical crystals of C_3A (Plate VI*a*), frequently with corners extended into sharp spears. Extensive areas of smaller square to rectangular crystals of C_3A, usually representing a succession of small cubes similar to the eutectic structure between C_3A and C_5A_3. The matrix of the whole structure is a low index formless material. No crystals of C_3S can be seen. (*b*) With crossed nicols (Plate VI*b*). The matrix proper is isotropic. The large cubes of C_3A show rather wide bordering zones with a steel gray birefringence, exceedingly finely fibrous in character. Narrower zones border all of the smaller C_3A crystals. The birefringent zones often yield good interference figures, all uniaxial and positive. The large cubes are not perfectly isotropic, but show a very faint plaid birefringence quite similar to that of leucite. No C_3S crystals could be observed. Within the large cubes there are found rounded grains of free CaO again serving as crystal nuclei.

Interpretation: Structures are rather more difficult to decipher than in the preceding charge. It is evident that at the burning temperature the liquid composition had gone beyond point 17; free CaO was present as a solid phase, though the C_3A may not have been completely melted. On the other hand, no silicate mineral could have been present as a solid phase. With cooling, the composition of the liquid probably returned through points 17 and 16. The complete absence of the usual easily recognizable forms of C_3S and C_2S makes the precise path a little difficult to follow. The tendency of C_3A to form large crystals is again in evidence.

The lack of C_3S and C_2S in the usual crystal forms is the outstanding observation. On the other hand, the birefringence exhibited definitely indicates that some silicate has crystallized. Since C_3A should form continuously through the whole sequence of cooling and crystallization, the obvious conclusion is that the C_3A and the silicates crystallize intimately intermixed, which is again indicated by the faint plaid birefringence within the large C_3A cubes, but which silicate it is difficult to say. Optically positive figures indicate C_2S.

The birefringent border zones are similar in some respects to the alteration borders on C_3S, previously described. If this is the case, optically positive figures would result, and a limited isomorphous crystallization (that is, a limited solid solution) of C_3A and C_3S is indicated. The only obvious conclusion is that some factor interferes with the separate crystallization of C_3A and the silicates, a factor not yet fully understood.

Specimen 3.—Mix: C_3A, 50 per cent, C_2S, 50 per cent. Heat treatment: 1 hr. at 1480 C.; 35 min. at approximately 1530 C.; steadily cooled through 35 min. to 1370 C. Thoroughly sintered but did not slump.

OBSERVATION: (*a*) With plane polarized light: A "spotty" structure, large patches of C_3S and C_2S grains segregated from one another (Plate VII*a*). The C_3S is in well-formed crystals and the C_2S in rounded grains. The matrix is not of uniform refractive index; in the C_3S areas the index is that of C_3A; in the C_2S areas it is distinctly lower, approaching that of C_5A_3. Also in the C_2S areas there is seen a rather open matted structure of narrow laths intersecting at right angles; these laths have the lowest index observed and are isotropic. (*b*) With crossed nicols: The field appears quite similar to that of many commercial clinkers, showing the C_3S areas in dark steel gray and the round C_2S grains with considerably brighter birefringence. The matrix is everywhere isotropic. The most notable observation is that of the distinct alteration borders of C_2S on the C_3S grains (Plate VII*b*).

INTERPRETATION: Stable liquid should form in this mix at 1455 C. At the maximum burning temperature the liquid composition was above point 16, on the boundary between C_3S and C_2S, which were the stable solid phases represented in the segregated areas observed. With cooling, C_3A could not form until the liquid composition reached point 16. This is an invariant point, and C_3A could not crystallize without addition of lime, which comes from alteration of C_3S. Since no C_3A could form prior to point 16, none of the C_3S was isolated and it all shows alteration borders.

It is through this necessity for extra lime that C_3A crystallized only in the C_3S areas. The continuous character of the C_3A through these areas again evidences the tendency of C_3A to crystallize in large units; rarely can any crystal outlines be seen. In the C_2S areas there is nothing to supply the lime deficiency; hence, the liquid composition moved down the quadruple line toward point 15. The right-angled lattice structure in the C_2S areas is made up of laths of C_5A_3, which indicates a beginning crystallization at point 15. The matrix in the C_2S areas is distinctly higher in index than these laths, but considerably too low for C_3A; hence, it is concluded that the matrix here is principally glass, with intermediate composition and refractive index.

Specimen 4.—Mix: C_3A, 70 per cent, C_2S, 30 per cent. Heat treatment: 1 hr.

at 1480 C.; 35 min. at approximately 1530 C.; steady cooling through 35 min. to 1370 C. Charge melted to complete slump.

OBSERVATIONS: (*a*) With plane polarized light (Plate VIII*a*): Large square crystals of C_3A are seen, enclosing grains of free CaO and crystals of C_3S. The C_3A squares usually show small clear areas in the center, but the principal part of the crystal shows a distinct fibrous structure, dividing the crystal into quadrants, with the fibers oriented perpendicular to the sides of the square. The fibers or stringers are observed to have a refractive index slightly higher than the C_3A. Plenty of clear material occurs as a matrix between the squares; its refractive index is considerably lower than the C_3A. (*b*) With crossed nicols (Plate VIII*b*): The fibrous areas exhibit a bright mass birefringence; interference figures are not difficult to obtain, especially in areas where the section happened to cut the fibers at right angles, where they then appear as dots; the figures are uniaxial and positive. Within the fibrous areas and in the matrix, the C_3S crystals are seen to be more or less completely altered to C_2S, yielding quite perfect pseudomorphs; within the clear central portions of the squares, however, the C_3S crystals show the usual low gray interference color, with no trace of alteration borders. The matrix is essentially isotropic, though it shows a very faint plaid birefringence. Within the matrix occasional bluntly radiate grains of C_2S can be seen.

INTERPRETATION: This mix is located on the high lime side of the line 17—C_3S; hence, heating above 1470 C. will result in the liberation of some free CaO as a solid phase. At higher temperatures the liquid composition will move up the CaO-C_3S boundary, but evidently the temperature attained did not melt all of the CaO or C_3S. On cooling, the liquid composition returned to point 17, where C_3A began to crystallize; as usual, the CaO grains served as nuclei, and the C_3A crystallized around the C_3S grains as well. This initial C_3A crystallization shows as the clear central areas, and both the C_3S and CaO enclosed were isolated from further reaction. With further cooling, the liquid composition moved to point 16, where C_2S should appear as a solid phase, and C_3S must decompose as C_2S and C_3A are formed.

Again there is observed a difficulty in the separate crystallization of C_3A and C_2S; the cubes of C_3A continue to grow, but C_2S crystallizes intimately with it, yielding the fibrous birefringent areas observed, and in these the C_3S crystals show heavy alteration borders. Similar fibrous birefringent structures are occasionally seen in commercial clinker, with compositions low in lime and high in alumina.

Specimen 5.—Mix: C_3A, 70 per cent, C_3S, 30 per cent. Heat treatment: 1 hr. and 20 min. at 1450 C. Withdrawn from furnace at that temperature. Good shrinkage obtained but no evidence of fusion or slump. Well sintered.

OBSERVATIONS: (*a*) With plane polarized light (Plate IX*a*). A continuous formless mass of clear C_3A. Combination of lime is nearly complete; there remains only a peppering of minute rounded grains. There is a similar peppering of minute irregular flakes of C_5A_3. The few dark angular objects are vacuoles representing part of the original intergranular space of the charge; angular outlines indicate lack of fusion. C_3S cannot be discerned; a few irregular small patches of slightly higher index (corresponding to C_2S) can be discerned with difficulty. (*b*) With crossed nicols (Plate IX*b*): An isotropic groundmass literally filled with

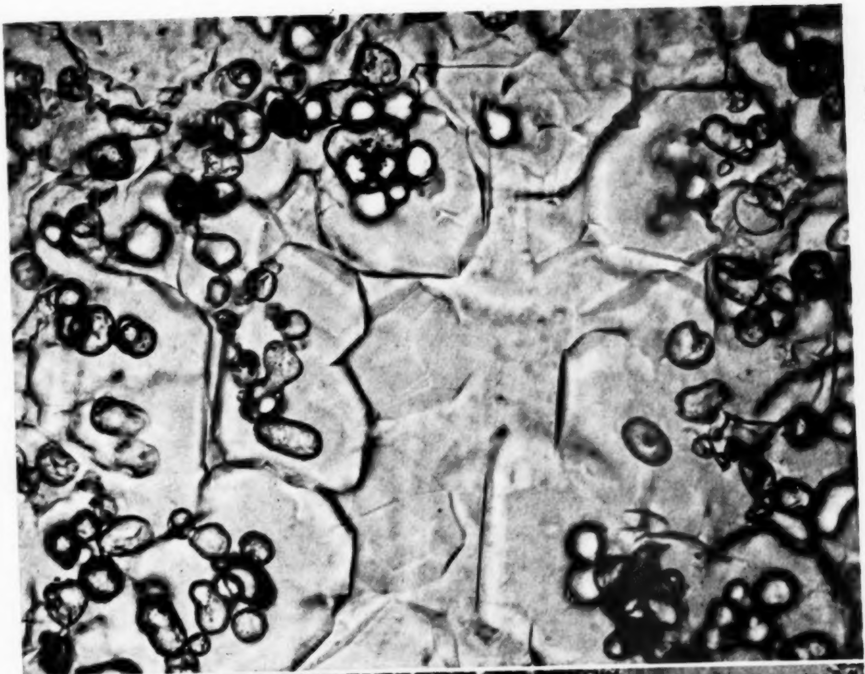


PLATE II.—Tricalcium Aluminate Heated to Dissociation and Recrystallized (× 300).

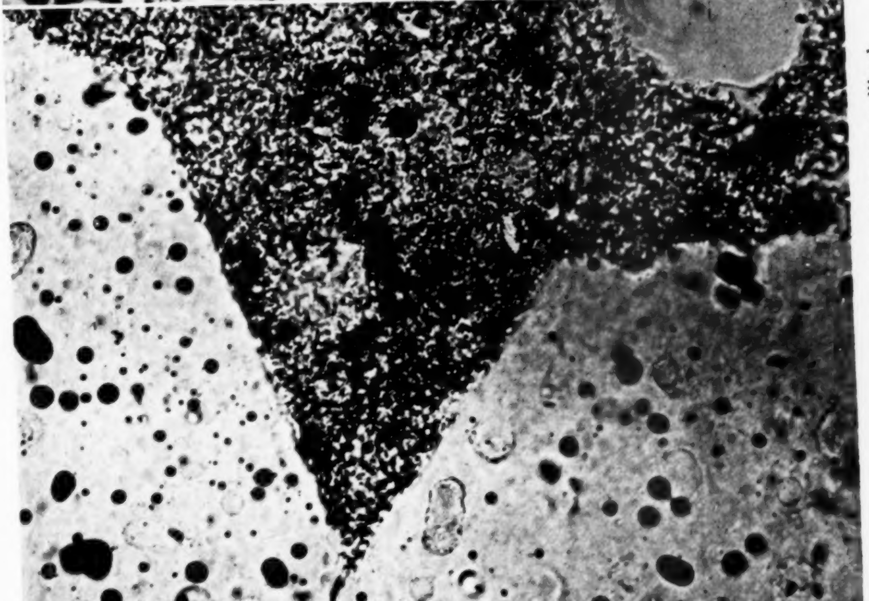


PLATE I.—Tricalcium Aluminate Partially Recrystallized at 1520 C. (× 300).

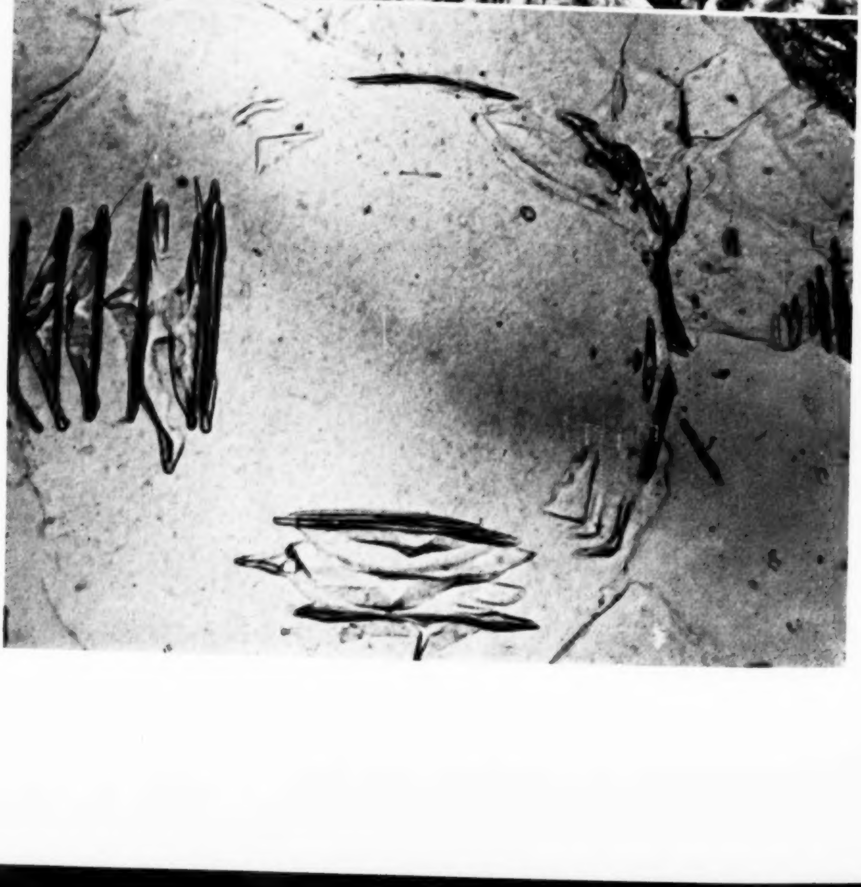


PLATE III.—C_3A, 35 per cent; C_5A_3, 65 per cent.
Phenocryst of C_3A (× 150).

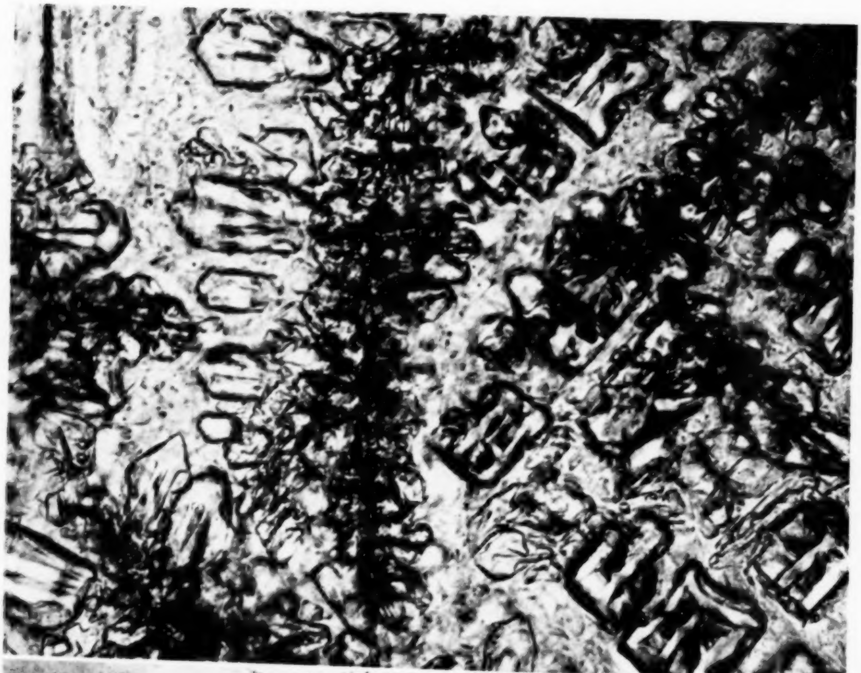
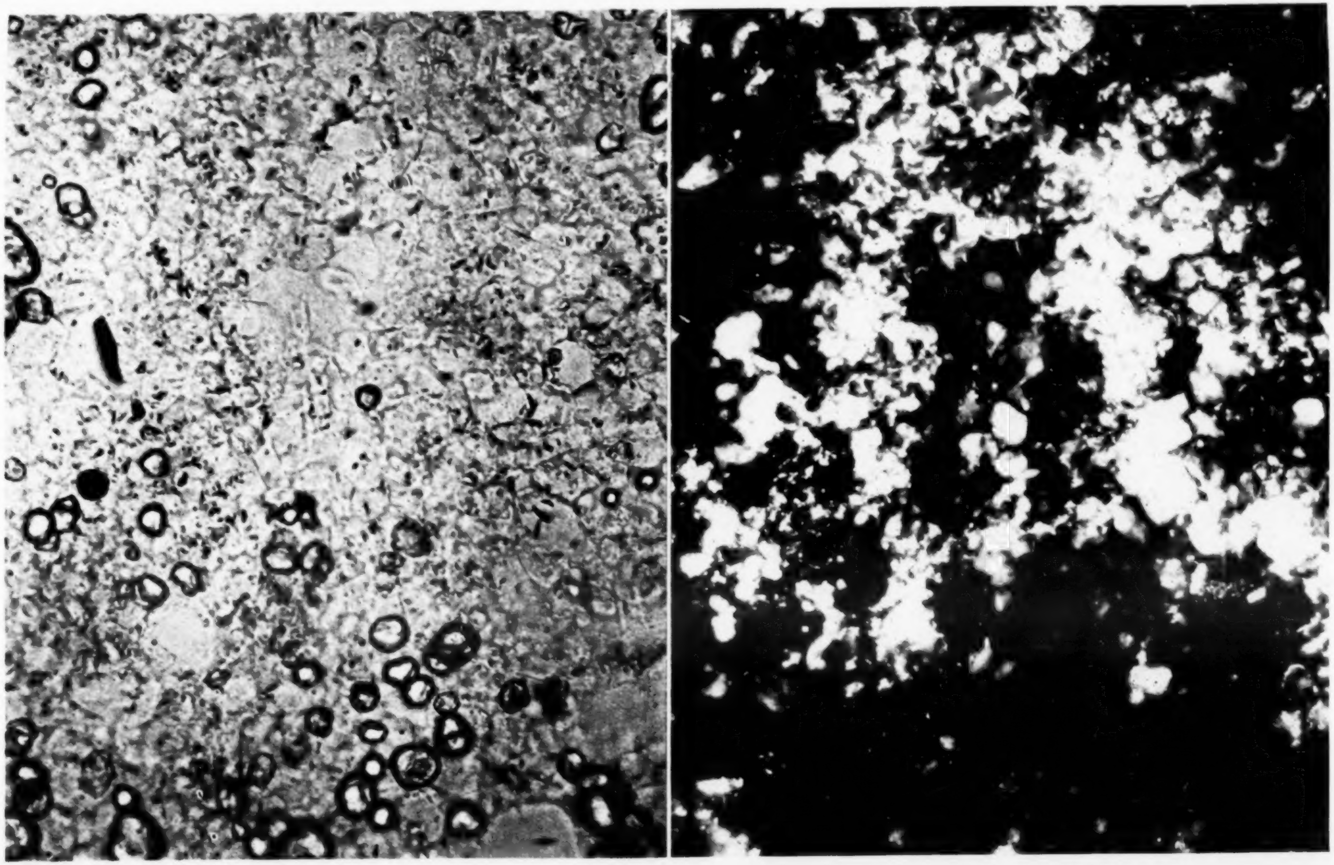
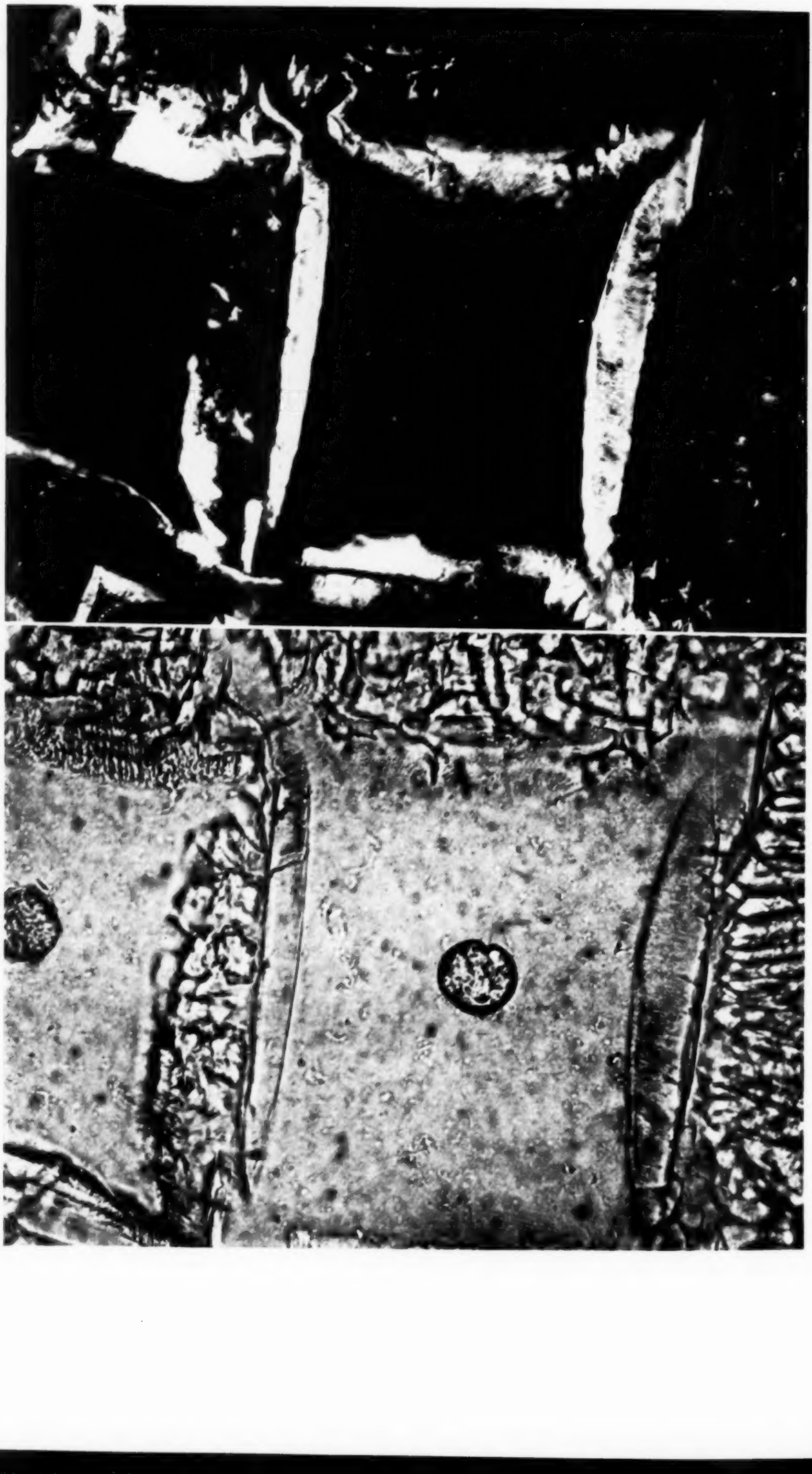


PLATE IV.—C_3A, 35 per cent; C_5A_3, 65 per cent.
Structure of eutectic (× 300).



(*a*) Plane polarized light (× 300). (*b*) Crossed nicols (× about 240).

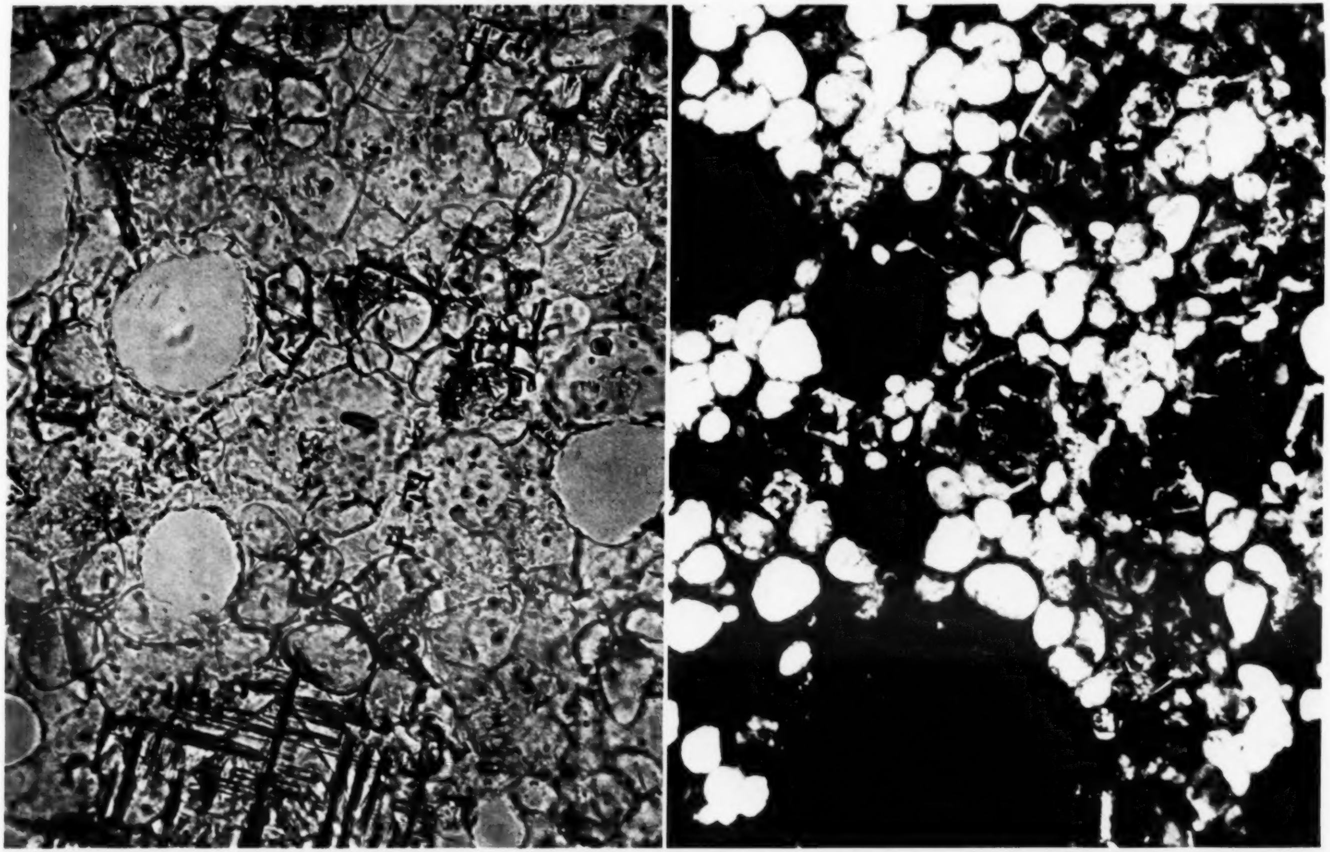
PLATE V.—C_3A, 40 per cent; C_3S, 60 per cent. Cooled from above 1470 C.



(a) Plane polarized light (× 300). (b) Crossed nicols (× about 240).

PLATE VI.—C_3A, 80 per cent; C_3S, 20 per cent. Cooled from above 1470 C.

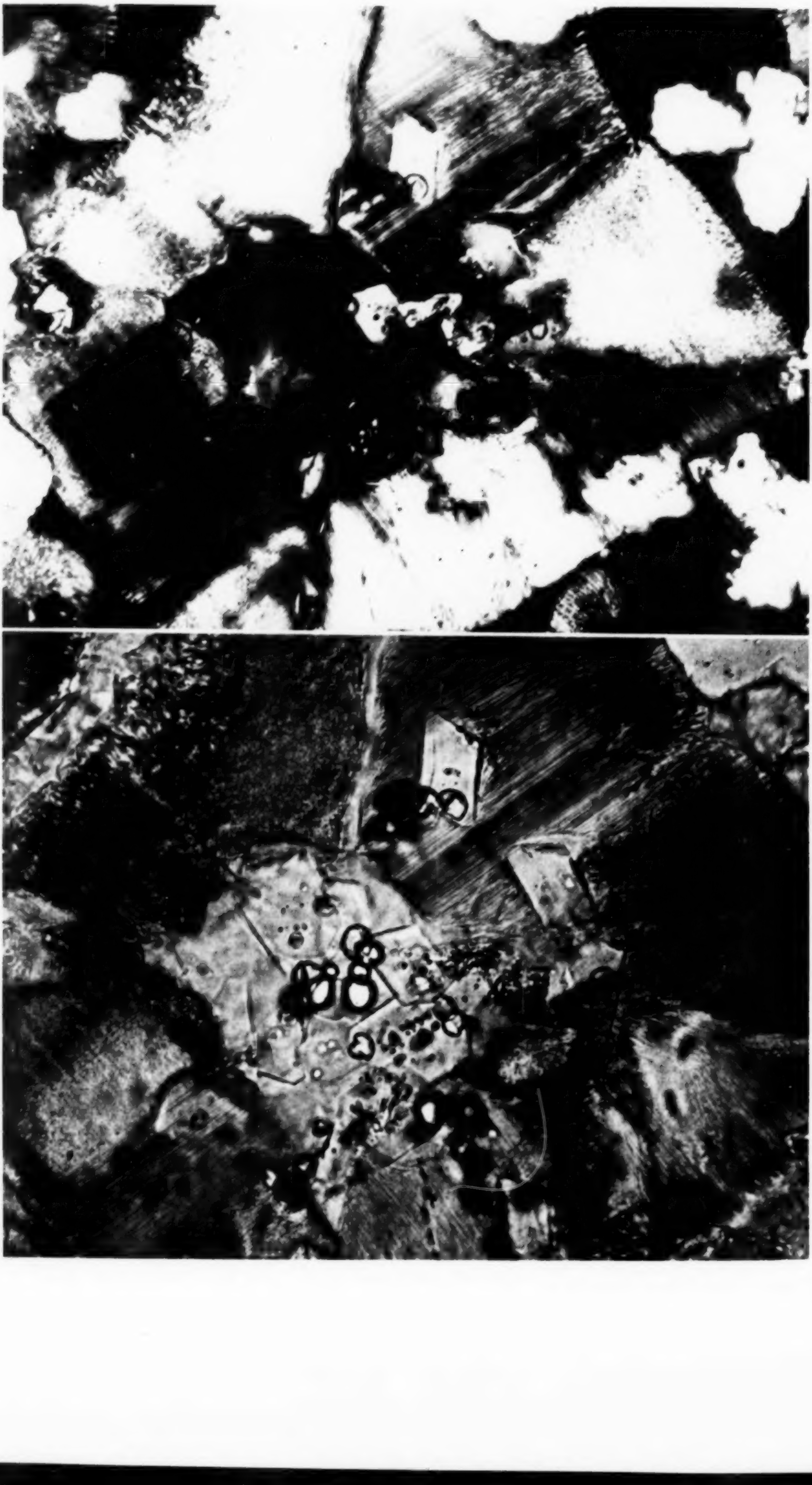
PLATE VI.—C_3A, 80 per cent; C_3S, 20 per cent. Cooled from above 1470 C.



(*a*) Plane polarized light (× 300).

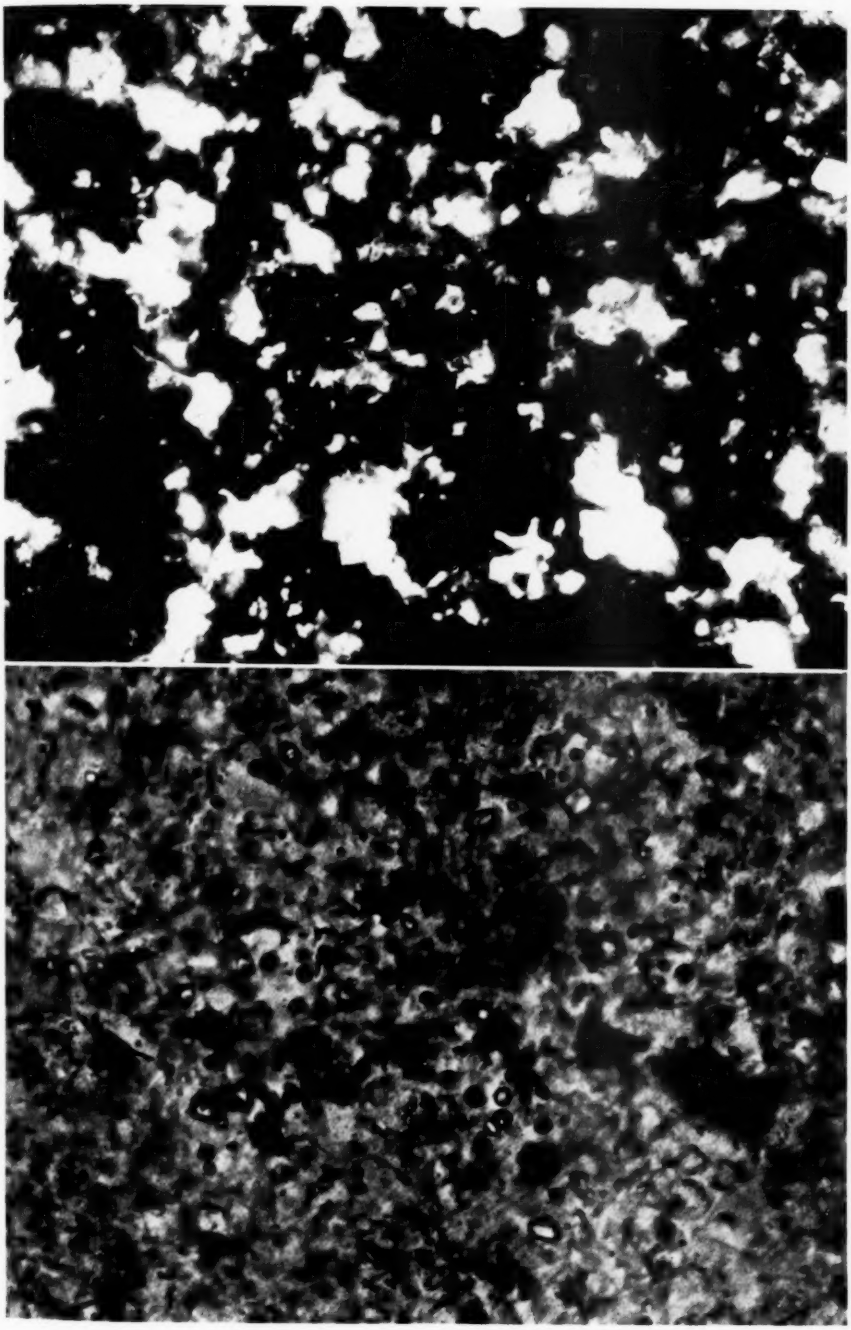
(*b*) Crossed nicols (× about 240)

PLATE VII.—C_3A, 50 per cent; C_2S, 50 per cent. Cooled from above 1470 C.



(a) Plane polarized light (× 300). (b) Crossed nicols (× about 240).

PLATE VIII.—C_3A, 70 per cent; C_2S, 30 per cent. Cooled from above 1470 C.



(*a*) Plane polarized light (× 600).

(*b*) Crossed nicols (× about 480).

PLATE IX.—C_3A, 70 per cent; C_3S, 30 per cent. Heated below 1470 C.

irregular shreds and flakes of C_2S and C_3S. No crystal outlines visible for either.

INTERPRETATION: There is little to be said in the matter of interpretation. Most noteworthy is the nearly complete combination effected, though in the structure developed it is almost impossible to visualize the relationships pictured in the phase equilibrium diagram. Probably the tendency of C_3A to form over large units is again the observed fact; the groundmass is continuous and no traces of single crystals can be perceived.

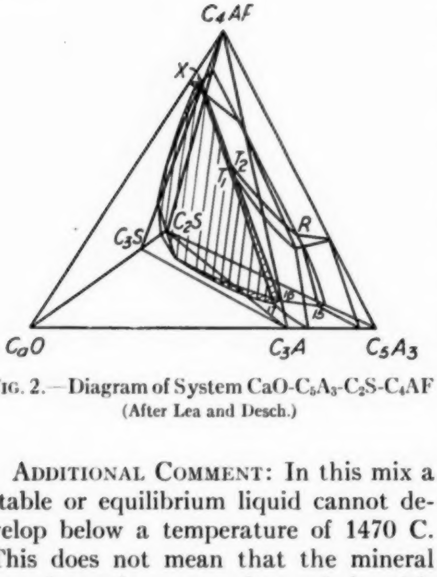


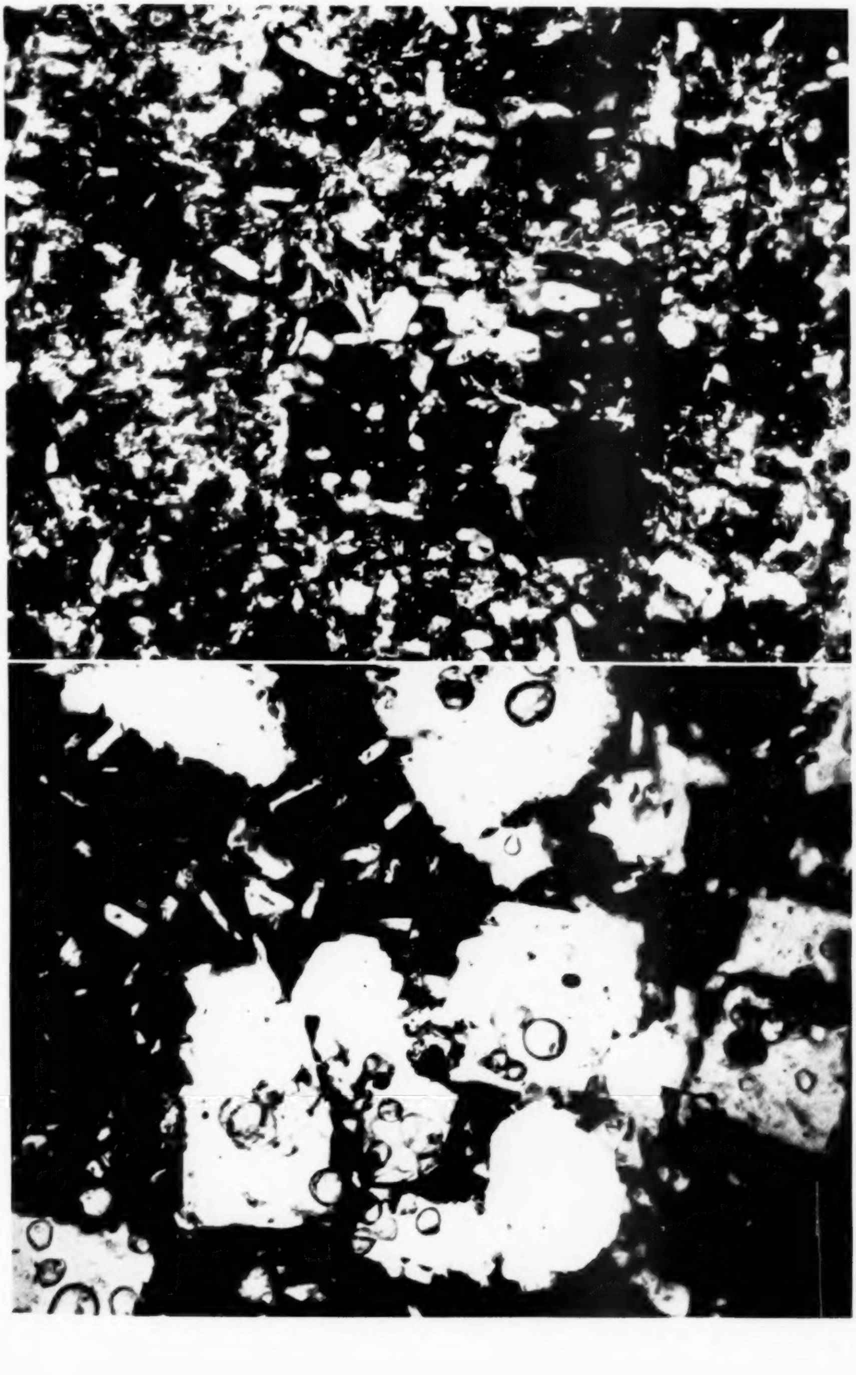
FIG. 2.—Diagram of System CaO-C_5A_3-C_2S-C_4AF (After Lea and Desch.)

ADDITIONAL COMMENT: In this mix a stable or equilibrium liquid cannot develop below a temperature of 1470 C. This does not mean that the mineral transformations, as observed in this section of a charge burned below 1470 C., were those of "solid reaction." Probably some liquid was formed at the lower burning temperatures, of composition represented by point 15, or even point 16. It must be pointed out that such liquids are not equilibrium liquids; they must be very transitory, both in amount and duration of their existence. Despite their doubly transitory nature, they probably are effective in promoting combination, and the true limits between solid and liquid reaction cannot be defined. It is also probable that reaction through this transitory liquid is an important factor in sintering.

C_3A IN THE QUATERNARY SYSTEM, CaO-C_5A_3-C_2S-C_4AF

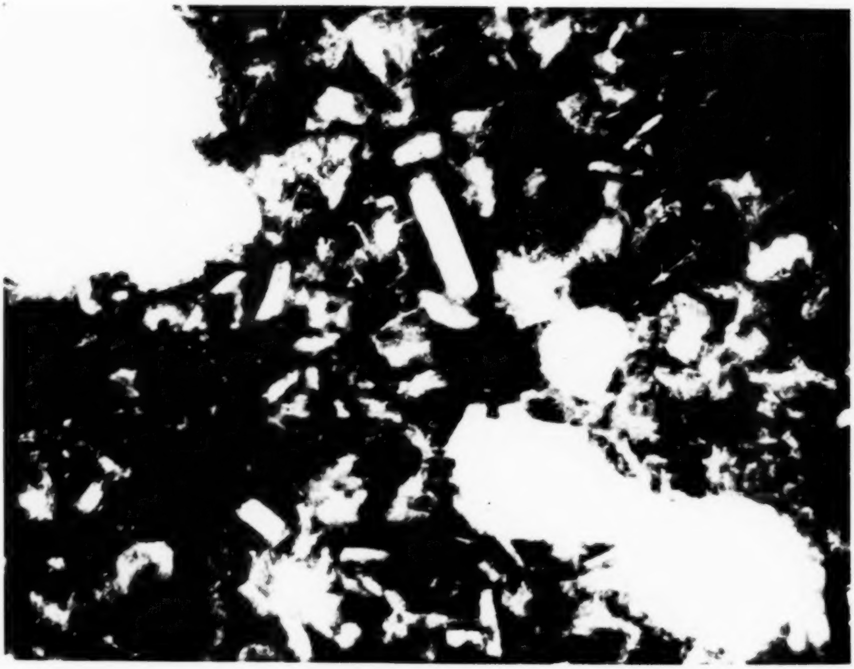
Figure 2 presents a diagram of the quaternary system as illustrated by Lea and Desch (5), with the C_3S primary phase volume shaded to facilitate visualization of spatial relations. Points T_1 and T_2 correspond to points 17 and 16 of the ternary diagram just considered, seen as the base of the tetrahedron. Portland-cement compositions occur within or on the high-lime side of the C_3S volume in a zone about three-fourths of the distance from T_1 to C_3S. Point T_2 is very close to the plane, C_3A-C_2S-C_4AF, and apparently just on the high-lime side of that plane, since the composition of T_2, as given by Lea and Parker (4, 6), calculates to 1.6 per cent C_3S. Thus T_2 is a eutectic, representing the termination of crystallization for any portland cement composition of the pure oxides lying on the low-lime side of the plane C_3A-C_3S-C_4AF. For any mix in or on the high-lime side of this plane, T_1 is the final crystallization point. Minimum temperatures of stable liquid formation are 1338 C. and 1341 C. at T_2 and T_1, respectively.

The alumina - ferric oxide ratio of T_1 is 1.38 (T_2 is essentially the same). On cooling the melt of a mix higher in ferric oxide, C_4AF will appear prior to C_3A, while from a mix higher in alumina the reverse is the case. If the mix is sufficiently high in lime, the course of the liquid composition, on cooling, will be in part along the quintuple lines 17-T_1, or x-T_1, with T_1 in each case the direction of falling temperature. With alumina in excess of the ratio 1.38, the point where C_3A begins to

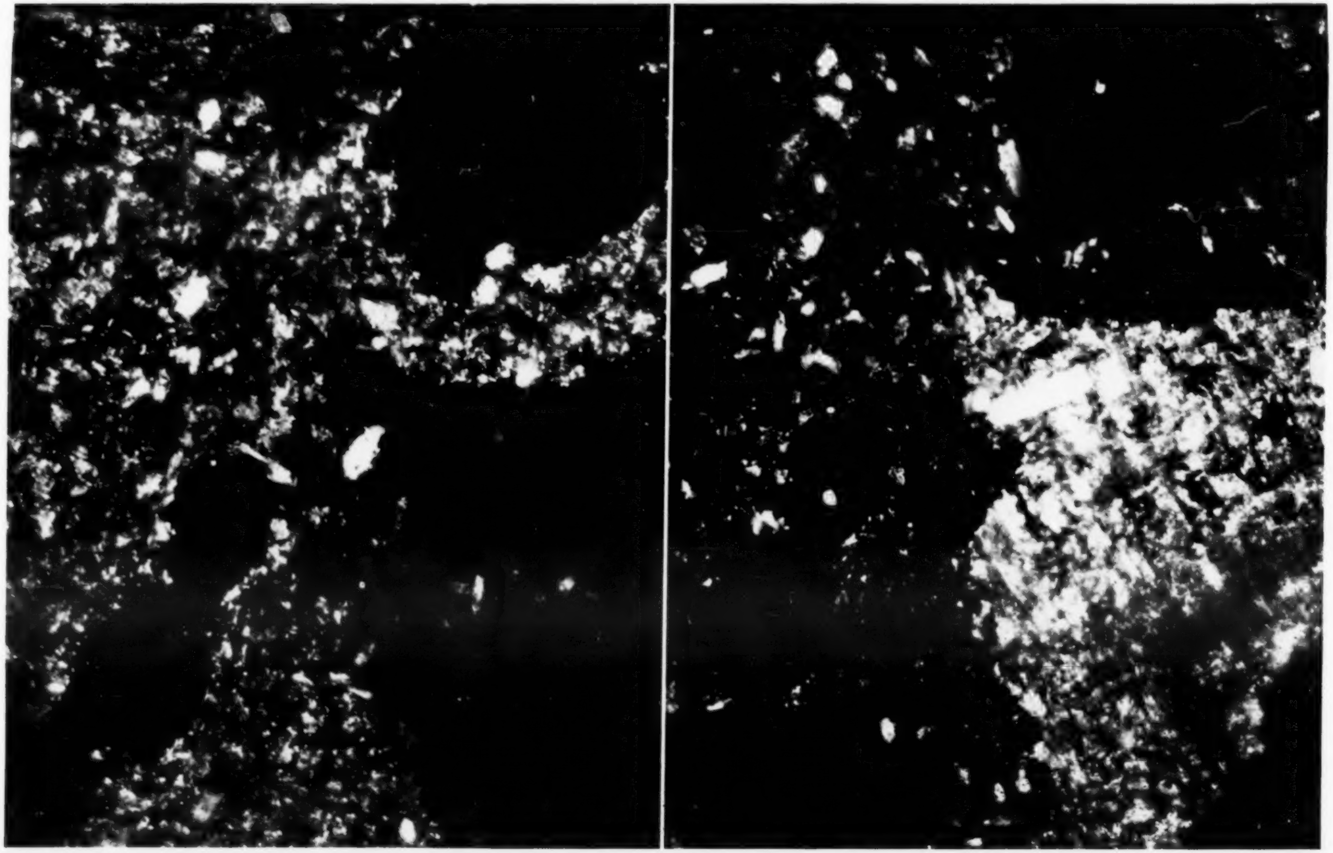


(a) Plane polarized light (× 300). (b) Crossed nicols (× about 240).

PLATE X.—C_3A, 23.0 per cent; C_4AF, 29.1 per cent; C_3S, 45.6 per cent; C_2S, 2.3 per cent. Cooled from 1420 C.



(a) Plane polarized light (× 300).



(*b*) Crossed nicols. C_4AF area at extinction (× about 240).

(*c*) Crossed nicols. Nicols 45 deg. from (*b*). C_4AF at maximum (× about 240).

PLATE XI.—Same Mix as in Plate X. Air-quenched at 1400 C.

crystallize will be somewhere between 17 and T_1. Such a mix may be chosen for illustration, since the chief interest in this paper concerns the behavior of C_3A:

Oxides, per cent	Potential Compound Composition, per cent	Alumina-Ferric Oxide Ratio
CaO . . 62.79	C_3S . . 45.58	
Al_2O_3 . . 14.82	C_2S . . 2.29	1.55
Fe_2O_3 . . 9.59	C_3A . . 23.00	
SiO_2 . . 12.80	C_4AF . . 29.13	

Heat Treatment: 40 min. at 1240 C.; 1 hr. at 1420 C.; steadily cooled through 20 min. to 1330 C.

Observations: (*a*) With plane polarized light (Plate X*a*): Large clear sharp cubes of C_3A, without octahedron, frequently enclosing rounded grains of free CaO. C_4AF, crystallized in a shredded or fern-like structure, outlines the cubes and encloses well-formed C_3S crystals. (*b*) With crossed nicols (Plate X*b*): The isotropic C_3A cubes are seen to contain numerous C_3S crystals. A few small flakes of C_2S can be discerned. The C_4AF shreds display mass birefringence around the cubes of C_3A, extinguishing when the sides of the cubes (edges of the squares in the section) are parallel to the vibration directions of the nicols. C_3S crystals do not show alteration borders, either within or without the C_3A cubes.

Interpretation: This mix contains a slight excess of CaO for the composition along the quintuple line at the point where the alumina-ferric oxide ratio is 1.55. The temperature of burning was such that all of the excess alumina was in the liquid, as will be shown presently, so that the liquid composition at that temperature was on the C_3S-CaO surface, and these two compounds were present as solid phases. With cooling, the liquid composition promptly reached the quintuple line, where C_3A began to crystallize, forming in large units enclosing both CaO and C_3S. The liquid composition then passed along the quintuple line, as C_3A continued to crystallize, eventually arriving at the invariant point T_1 where C_4AF appeared with C_3A, forming the matted intergrowth observed. T_2 shortly was reached, and a small amount of C_2S was formed, but apparently complete equilibrium crystallization was not attained, since the presence of free lime should markedly increase the amount of C_2S over that calculated (2.29 per cent) from the entire mix.

Additional Comments: 1. Presence of ferric oxide in an aluminous mix slightly raises the refractive index of C_3A. In the ternary system, lime-alumina-silica, the index of C_3A is always slightly lower than the resin (1.7135); as ferric oxide is added to the ternary mix, the index gradually increases; in the present mix (alumina-ferric oxide ratio, 1.55) the index is higher than the resin, and so nearly that of C_3S that crystals of C_3S in the C_3A cubes cannot be distinguished with ordinary illumination; they are seen only with crossed nicols. The indices observed agree with those given by Lea and Parker (6).

2. To show that the cubes of C_3A observed were produced by slow cooling, the same mix was heated to 1400 C. and quenched in air by removal of the charge from the furnace and placing it on a cold stone surface. The cubes are entirely lacking (Plate XI (*a*)). A small amount of glass was observed on the bottom, but for the most part the liquid has partially differentiated. Most interesting are the patches of right-angled lattice structure developed in the quickly cooled liquid. The same field is shown with crossed nicols in Plates XI*b* and XI*c*. There are large patches within which the crystallites of C_4AF are commonly oriented, resulting in a mass

birefringence. It is noteworthy that these areas are coextensive with the areas of lattice structure, as illustrated, and that they extinguish with the lattice parallel to the nicol vibration directions. Plates XI*b* and XI*c* differ from each other in a 45-deg. rotation of the nicols, to illustrate more clearly the mass extinction and areal boundaries.

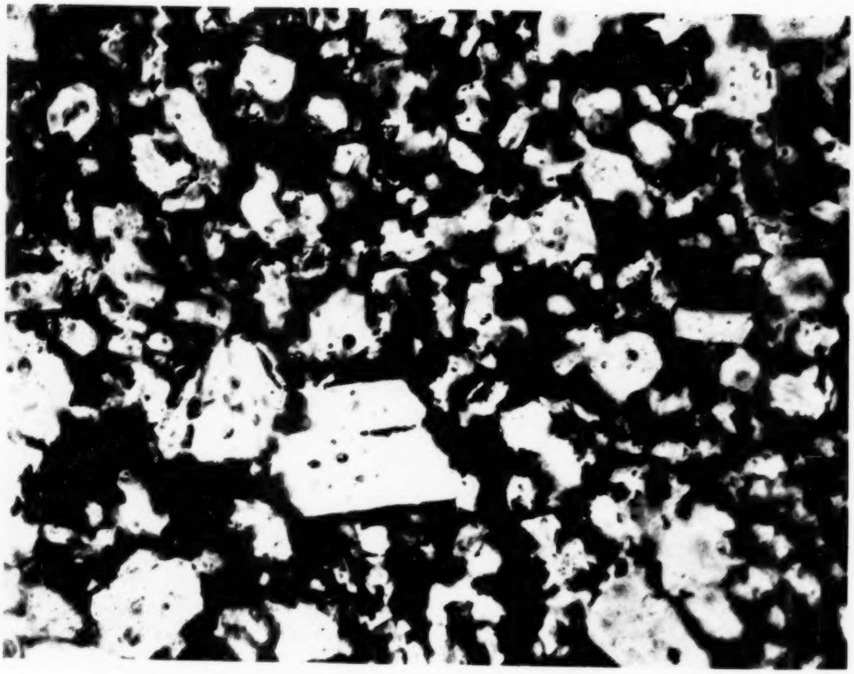
The common orientation of the C_4AF crystallites indicates the ease and rapidity with which C_3A tends to crystallize; in this case there was not time enough to permit segregation of compounds, but the C_3A rapidly reached out over wide areas in the form of skeletal crystals, governing the orientation of the C_4AF crystallites. Evidence supporting this conclusion is that (*a*) C_3A should crystallize from this mix prior to C_4AF; (*b*) the right-angled lattice structure indicates isometric crystallization; (*c*) the crystal habit of C_4AF is acicular or bladed (monoclinic); (*d*) the birefringent patches are usually equidimensional and occasionally quite perfect squares, as illustrated; (*e*) observed influence, previously mentioned, in slow-cooled specimens of the C_3A cube directions on extinction of contiguous C_4AF.

3. To determine compositions from which similar C_3A cubes could be produced, two series of mixes were studied, one with C_3S constant at 50 per cent and one at 75 per cent, the remaining 50 or 25 per cent being alumina and ferric oxide in varying ratios. Slow cooling produced cubes of C_3A in every case where the alumina-ferric oxide ratio was 1.55 or higher, an observation that indicates slight if any solid solution of C_3A in C_3S. No charges were prepared with alumina-ferric oxide ratio between 1.55 and 0.96, but the results obtained conform to the relations indicated in the quaternary diagram. In general, there is a better cube development in high-lime mixes. On the other hand, it should be pointed out that free lime is not essential to their formation; lower limed mixes develop cubes almost as easily. Sections showing free lime have been chosen for illustration only to extend further the visible portion of the course of crystallization.

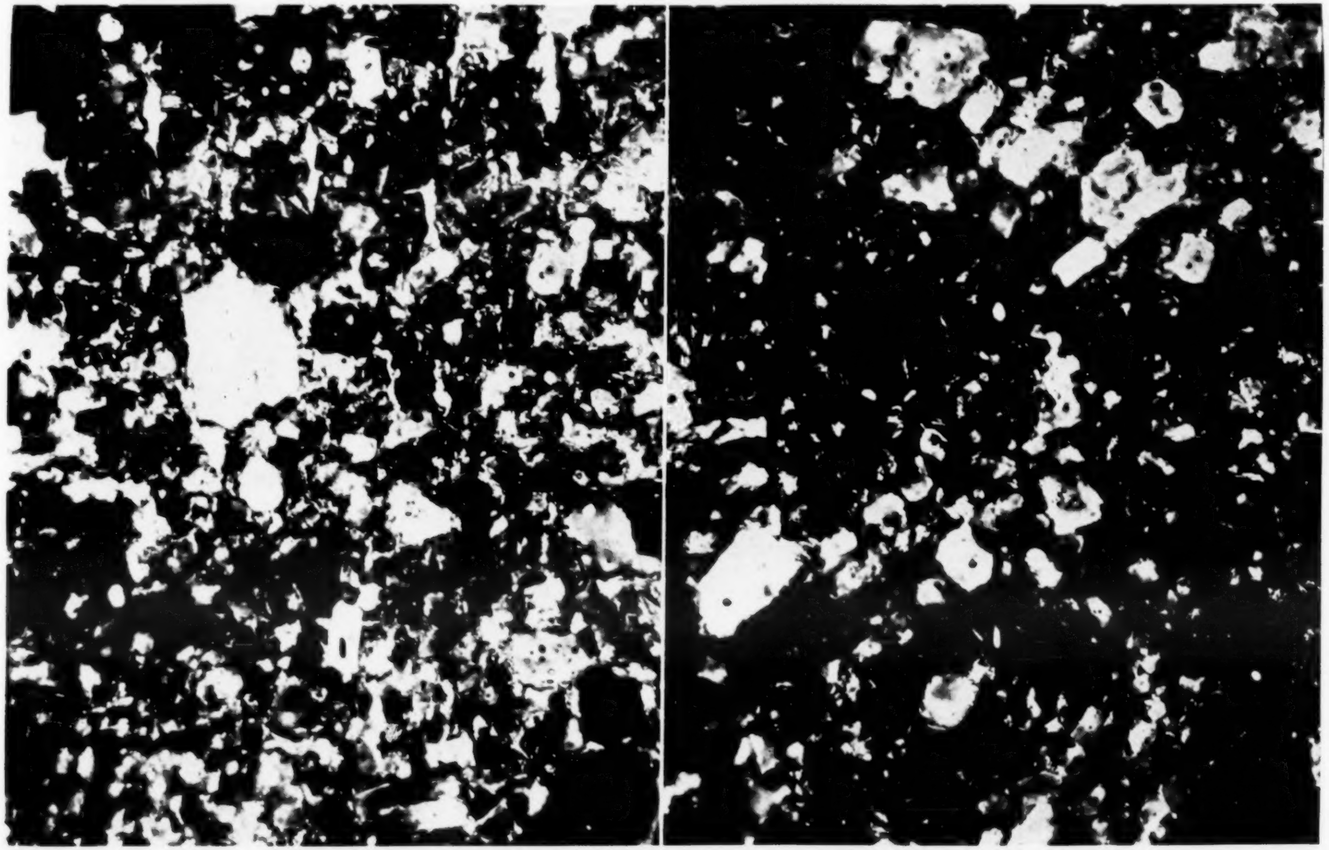
COMMERCIAL CLINKER

In the foregoing study of experimental charges, the intention has been to trace C_3A from high-alumina mixes into the range of commercial portland cement compositions, with especial reference to the structural relationships developed. It has been observed in the experimental mixes that C_3A crystallizes easily and rapidly, that it tends to form in large units, and that it tends to control the orientation of C_4AF when the latter follows C_3A in the course of crystallization, that is, mixes with alumina-ferric oxide ratios greater than 1.38. Thus the structural evidences of crystalline C_3A to be sought in commercial clinker are principally two: (1) large clear cubes of C_3A enclosing C_3S and possibly CaO, and (2) extended areas in which C_4AF crystallites possess a common orientation, exhibited as mass birefringence, or both.

In the experience of this laboratory, no commercial clinker has shown the cubes of C_3A. On the other hand, more or less extended areas of C_4AF mass birefringence are fairly commonly observed. For example, a section of commercial clinker C (below) is shown in Plates XII*a*, XII*b*, and XII*c*. In XII*a* the crystals of C_3S are sharply outlined against a partially differentiated glassy matrix; there is no hint of continuity or common orientation of the C_4AF crystallites in the matrix. With crossed nicols the C_4AF crystallites over the entire field light up and extinguish simultaneously. Plate XII*b* shows them at a maximum and

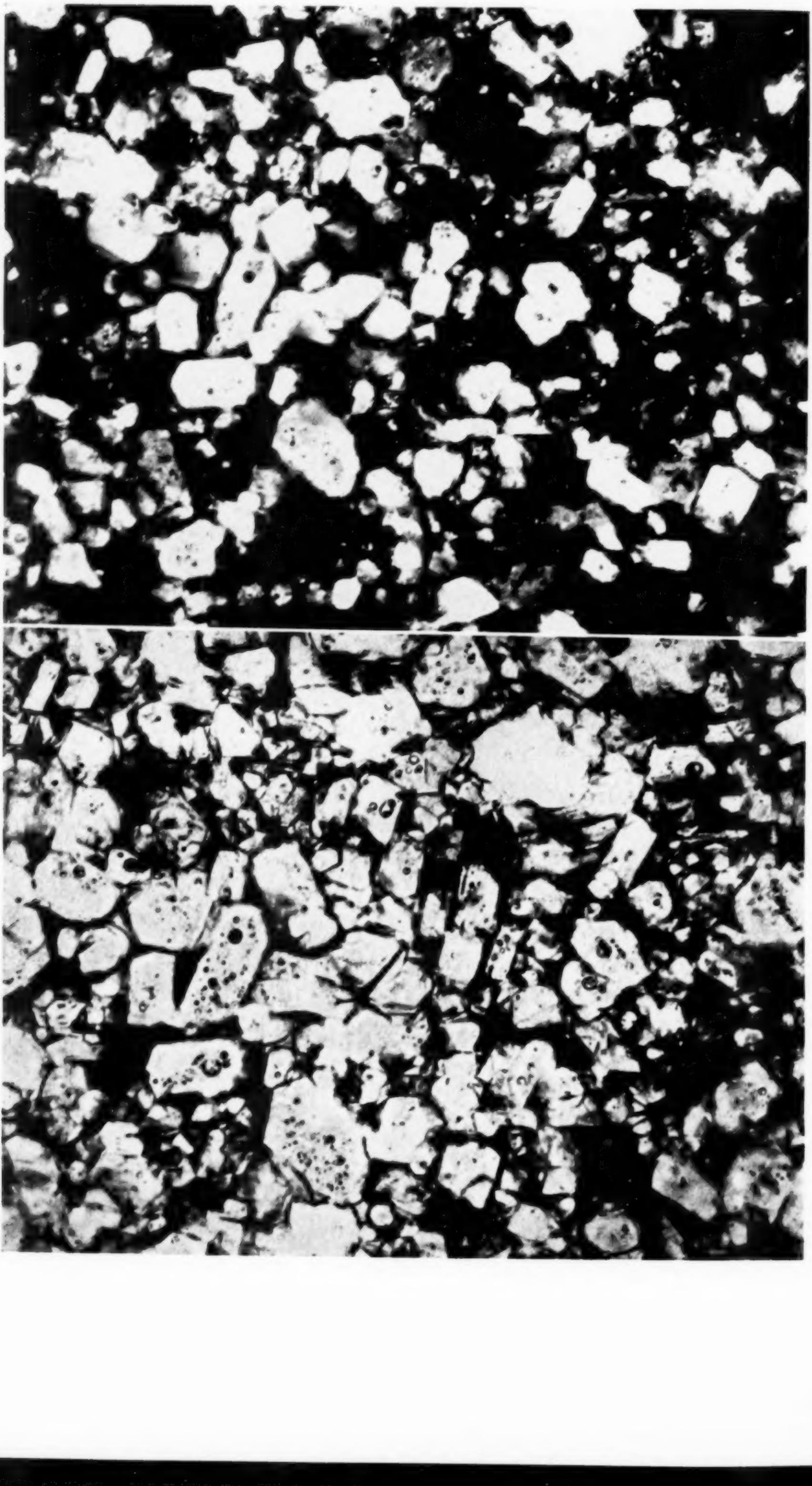


(a) Plane polarized light (× 300).



(*b*) Crossed nicols. C_4AF at maximum (× about 240). (*c*) Crossed nicols. Nicols 45 deg. from (*b*). C_4AF at extinction (× about 240).

PLATE XII.—Thin Section of a Commercial Clinker.



(a) Plane polarized light (× 300). (b) Crossed nicols. Glass is isotropic (× about 240).

PLATE XIII.—C_3S Crystals in Glass. Commercial mix air-quenched at 1340 C.

XII*c* at extinction (rotation of the nicols through 45 deg.). The striking nature of the effect is difficult to capture photographically, in the absence of the brilliant yellow color.

By moving the slide it is found that these large areas of common birefringence are roughly equidimensional; they illustrate again the coarse crystallization of C_3A, although no crystal outlines can be seen.

To determine whether clear cubes could be formed in commercial clinker, several experiments were performed using clinkers of commercial manufacture. The clinker analyses of the materials chosen are shown in Table I.

TABLE I.—CLINKER ANALYSES

	CLINKER A	CLINKER B	CLINKER C	CLINKER D	CLINKER E	CLINKER F	CLINKER G
SiO_2, per cent	19.32	21.56	19.92	22.80	22.00	21.29	22.56
Al_2O_3, per cent	7.02	4.25	7.17	6.70	6.08	6.08	6.48
Fe_2O_3, per cent	2.78	3.69	3.67	2.90	3.00	3.35	2.60
CaO, per cent	65.69	66.37	67.67	66.00	67.64	67.35	66.57
MgO, per cent	3.31	3.20	0.90	0.80	0.90	1.40	1.29
SO_3, per cent	0.80	0.58	0.35	0.44	0.10	0.10	0.02
Loss on ignition, per cent	0.04	0.20	0.25	0.17	0.15	0.02	0.37
Total	98.96	99.85	99.93	99.81	99.87	99.59	99.89
SiO_2-R_2O_3 ratio	1.97	2.72	1.84	2.37	2.42	2.26	2.48
Free CaO, per cent	2.10	0.48	1.40	0.36	0.52	0.30	0.24
Alumina - ferric oxide	2.52	1.15	1.95	2.31	2.02	1.81	2.49

The clinkers were ground to pass a No. 100 sieve; small charges (about 3 g.) were burned, and structures were observed, as indicated below.

Clinkers A, B, and C.—Heat treatment: 15 min. at 1500 C.; steadily cooled through $2\frac{1}{4}$ hr. to 1340 C. Well sintered but no evidence of slumping.

OBSERVATIONS: Well-formed, euhedral crystals of C_3S set in an abundant formless matrix. Clinkers A and C showed a little free CaO but no C_2S; clinker B showed a few grains of C_2S, not gamma form, but no CaO. The matrix in each case was isotropic, with practically no trace of differentiation—a more or less dark-colored glass. No cubes of C_3A or mass birefringence was exhibited. (C_4AF should precede C_3A in clinker B, but crystalline C_4AF was absent). Clinker C is shown with plane light and with crossed nicols in Plates XIII*a* and XIII *b*, respectively.

Clinkers A and B.—Heat treatment: 10 min. at 1520 C.; cooled steadily through $2\frac{1}{2}$ hr. to 1250 C. No slump, but well sintered.

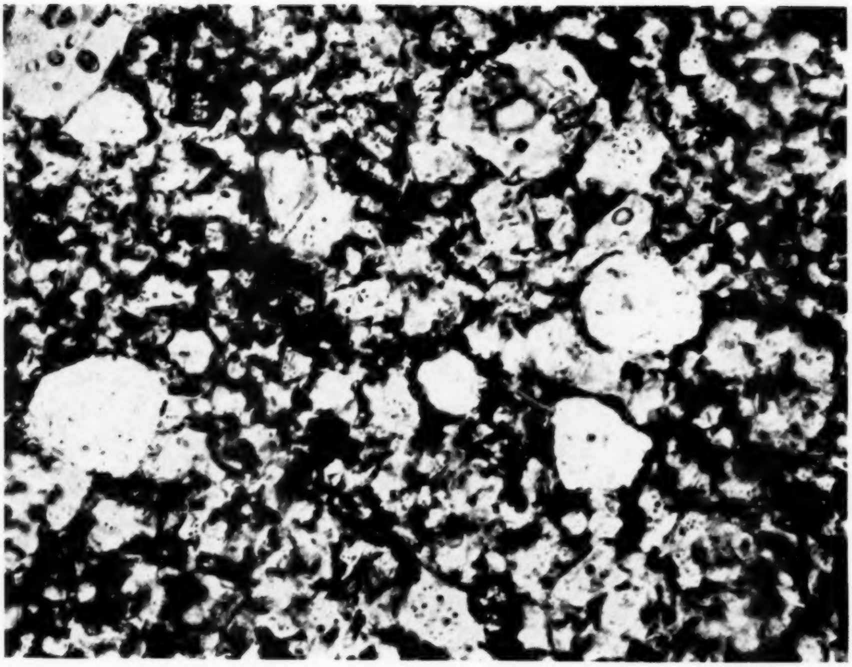
OBSERVATIONS: Euhedral crystals of C_3S, as before, except that discolored glassy matrix is absent. Some free CaO in clinker A; some C_2S in clinker B, not dusted. No cubes of C_3A have developed, although potentially about 13 per cent of C_3A should be present. Plate XIV*a* shows clinker A in ordinary light. The striking mass birefringence of the C_4AF crystallites is shown with crossed nicols, covering the entire field. In Plate XIV*b* they are at extinction, and in XIV*c* at a maximum.

Clinkers A and B.—Heat treatment: 15 min. at 1500 C.; steadily cooled through $1\frac{1}{4}$ hr. to 1180 C.

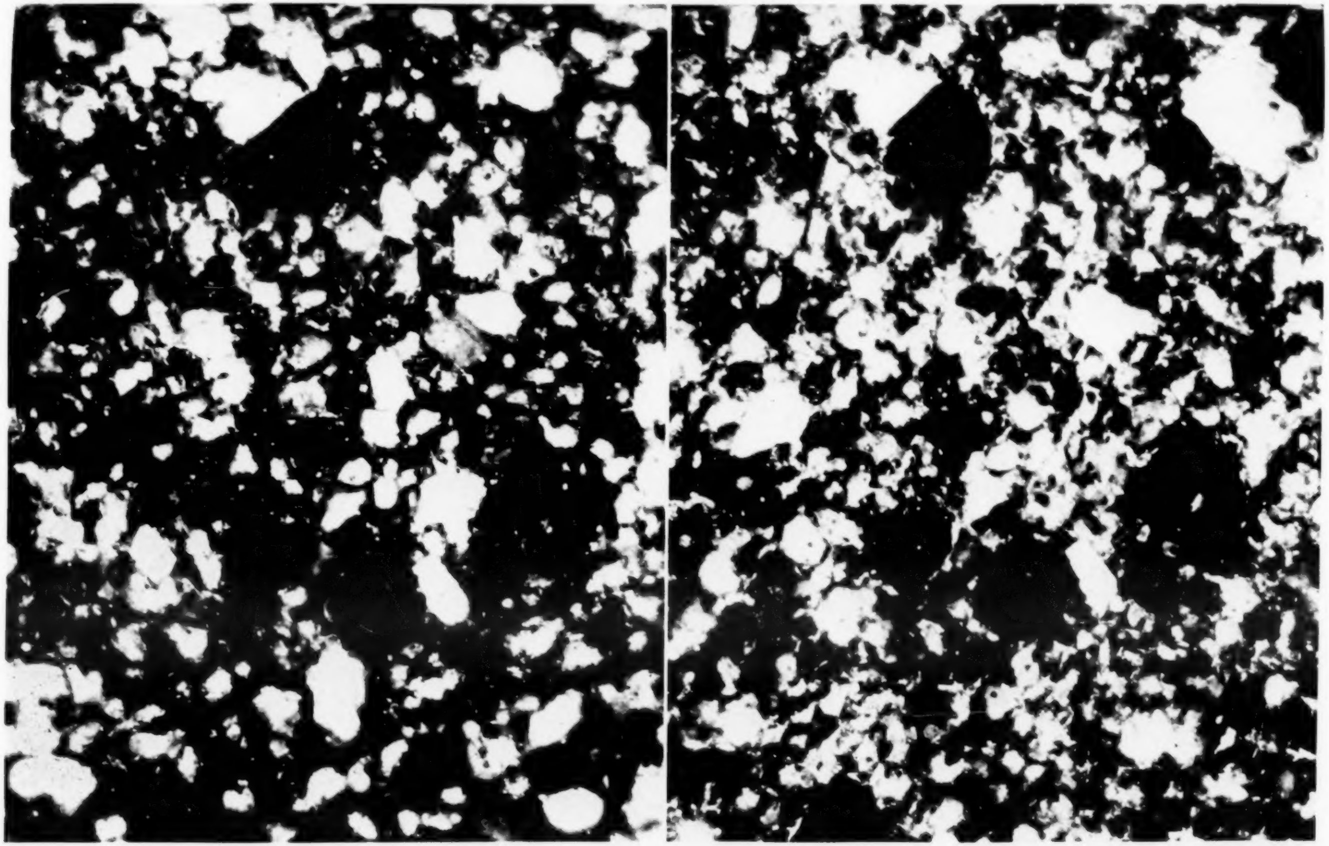
OBSERVATIONS: Both clinkers essentially the same as in the preceding. No dusting of C_2S.

Clinkers C and D.—Heat treatment: 15 min. at 1500 C.; steadily cooled through 55 min. at 1270 C. Well sintered but no slump.

OBSERVATIONS: Clinker C contained free CaO, clinker D a little C_2S, not dusted. No cubes of C_3A in either, but both showed excellent mass birefringence of C_4AF. Traces of glass remain in clinker C.

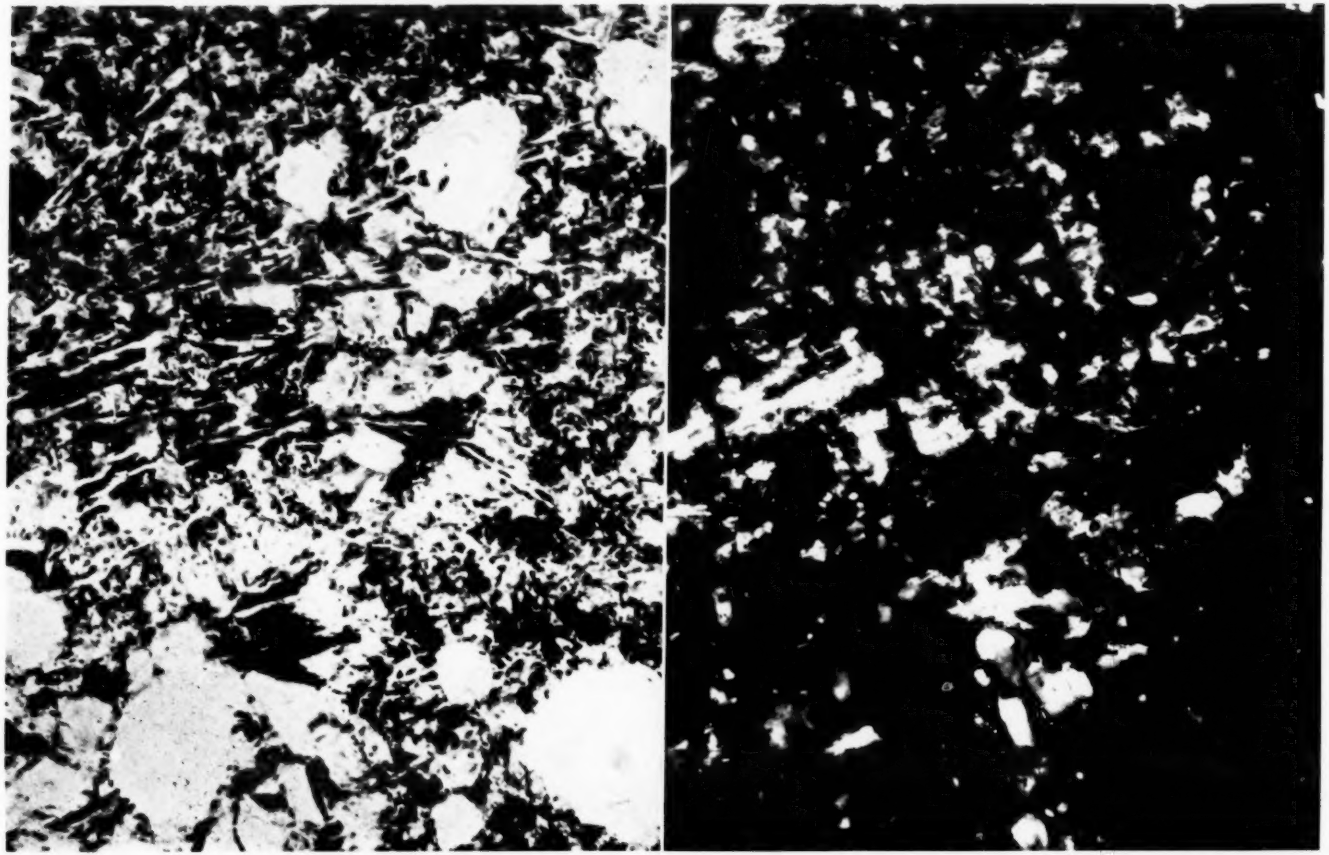


(a) Plane polarized light (× 300).



(*b*) Crossed nicols. C_4AF at extinction (× about 240). (*c*) Crossed nicols. Nicols 45 deg. from (*b*). C_4AF at maximum (× about 240).

Plate XIV.—Commercial Clinker High in Potential C_3A. Cooled slowly from 1520 to 1250 C.



(*a*) Plane polarized light (× 300). (*b*) Crossed nicols (× about 240)

PLATE XV.—Bladed Structure in Clinker High in Potential C_3A.

Clinker D, two samples.—Heat treatment: 15 min. at 1500 C.; steadily cooled through ½ hr. to 1420 C., when one specimen was removed (*a*); cooling continued through 1 hr. to 1310 C. for other specimen (*b*).

OBSERVATIONS: (*a*) showed clear crystals of C_3S and C_2S (not dusted) separated by thin stringers of greenish glass, the latter exhibiting a mere beginning of differentiation. In specimen (*b*) the C_2S has increased in amount, still not dusted, but it is clouded with sharp irregular inclusions; these could not be identified, but they apparently are discolored by some iron compound. Clear C_4AF occurs in segregated patches, showing continuous birefringence. Square outlines of C_3A cubes are not observed, but clear colorless areas are made up of several C_3S crystals, variously oriented but apparently crowded together, with interstitial material rarely evident. No traces of glass apparent.

Clinkers E and F.—Heat treatment: 10 min. at 1500 C.; steadily cooled through 1 hr. to 1280 C.

OBSERVATIONS: Apparently completely crystallized. No cubes of C_3A observed, but C_4AF shows continuous birefringence over large areas. C_4AF more abundant in clinker F, with higher ferric oxide content.

Clinker G.—This clinker was not crushed. The clinker was broken in half; one half was sectioned in the original condition, the other half heated as a single piece. Heat treatment: 10 min. at 1500 C.; steadily cooled through 45 min. to 1280 C.

OBSERVATIONS: The heated portion apparently was completely crystallized; no cubes of C_3A formed, but C_4AF shows well-developed mass birefringence. The latter was somewhat more definite in the heated half than in the original; otherwise, little difference was noted.

A peculiar structure commonly observed in commercial clinker should be described, a structure that is restricted to clinkers of rather high alumina-ferric oxide ratio and moderately high in lime. The structure reveals groupings of narrow clear laths separated by ragged stringers of C_4AF, the latter displaying mass birefringence over each group. The clear laths have the birefringence of C_3S, which, however, is discontinuous, so that the structure disappears with crossed nicols except for the mass birefringence of the C_4AF.

In laboratory burns a series of charges differed in alumina-ferric oxide ratios but with C_3S constant at 75 per cent. In the highest alumina-ferric oxide ratios the cubes of C_3A were well defined, but with intermediate alumina-ferric oxide ratios the same groupings of parallel laths appeared. The known composition and heat treatment, coupled with the observations previously recorded as to the behavior of C_3A, lead to a possible explanation. Take the following mix, for example:

CaO, per cent	68.08
Al_2O_3, per cent	8.90
Fe_2O_3, per cent	3.29
SiO_2, per cent	19.73
Alumina-ferric oxide ratio	2.70

Heat treatment: 40 min. at 1450 C.; steadily cooled through 20 min. to 1400 C. Well sintered but no slump.

OBSERVATIONS: This burn is illustrated with plane polarized light in Plate XV*a*; with crossed nicols in Plate XV*b*. The observations are those given in the description immediately above.

INTERPRETATION: Crystallization of C_3S from a rapidly cooled melt produces long narrow blades, as is well known. In this instance a mix high in potential C_3S and C_3A was rapidly cooled from a temperature of 1400 C. At this temperature, the liquid composition of the mix is within the C_3S volume or on the surface between C_3S and CaO, probably not far from the

PLATE XV.—Bladed Structure in Clinker High in Potential C_3A.

C_3A surface. Thus there is a rapid and simultaneous crystallization of C_3S and C_3A as the first phases to separate. The C_3S tends to confer a directional character to the crystallization of C_3A, although the actual crystallization of C_3S takes place about the borders of previously existing C_3S grains variously oriented. The clear blades thus are essentially crystals of C_3A, controlling the orientation of the shortly subsequent C_4AF crystallites in between. The relative concentration of C_3S as compared with C_3A tends to crowd the C_3S grains together, so that no part of the blades is perfectly isotropic, although the birefringence of the C_3S is discontinous. The influence of rapid crystallization in this case is shown by the fact that the parallel lath groupings described were found toward the base of the charge, the zone more rapidly chilled, whereas higher up in the charge a square development was observed, with somewhat better crystallization of C_4AF.

SIGNIFICANCE: In the absence of an understanding of the crystal habit of C_3A, it is difficult to correlate this structure with the phase equilibrium diagram, and the conclusion might be drawn that a new phase had appeared. The explanation offered shows no departure from equilibrium phases diagrammed. More importantly, the structure evidently develops through rapid cooling from moderately high temperatures, thus indicating only partial differentiation of the liquid phase.

CONCLUSIONS

1. The tendency of C_3A to crystallize rapidly in large units, enclosing crystals of C_3S, and crystallizing with especial ease around grains of free CaO, confers upon crystalline C_3A an apparent formlessness, so that in a completely crystalline clinker the crystalline C_3A becomes the real "interstitial" material. In commercial clinker the ratio of potential C_3S to potential C_3A is high; thus the C_3S crystals are crowded closely together, and the sparse interstitial envelope of crystalline C_3A is exceedingly difficult to distinguish. The difficulty is enhanced by the isotropism of C_3A, its clear and colorless character, and the close approach in refractive indices between C_3S and C_3A in ferriferous charges. Two other possible factors adding to the difficulty should be mentioned: (*a*) It has been shown that C_3A separates with difficulty from the silicates, under certain conditions, and the resulting C_3A exhibits a birefringence similar to C_3S. (*b*) If solid solution of C_3A in C_3S is possible to an appreciable extent, the volume of the interstitial envelope will be further decreased. Limits to this possibility have not been precisely established in this work, but it has been shown that the possible extent is not large.

2. Indirectly, the presence of crystalline C_3A in a clinker with alumina - ferric oxide ratio above 1.38 is demonstrated by the existence of any crystalline C_4AF, and especially plainly indicated where C_4AF shows continuous birefringence over large areas. The latter of course cannot be easily perceived in powder mounts (cements), and it is not well evident in sections, unless unusually thin.

3. In experimental mixes of the pure oxides, the structures developed can be correlated easily with the quaternary phase equilibrium diagram mapped by Lea and Parker (**6**). Difficulties appear only in underburned charges or those quickly cooled but not quenched. The lack of alteration borders of C_2S on C_3S crystals in the quaternary system confirms Lea and Parker (**4, 6**) in their location of point T_2 on the high-lime side of the plane, C_3A-C_2S-C_4AF, thus making T_2 a eutectic.

4. Observations on the behavior of commercial clinker indicate some departure from the strict conditions out-

lined by the quaternary diagram, though the general course of the reactions is the same. Thus, in clinkers A, B and C described above neither C_4AF nor C_3A had formed from the liquid on cooling to 1340 C.; the only solid phases were C_3S and C_2S, or C_3S and CaO. Ultimate crystallization apparently occurs in the vicinity of 1280 to 1300 C., agreeing with the observations of Hansen (7). This, however, does not seem to be related to the quaternary eutectic at point *R*, where the temperature is 1280 C., since no quaternary experimental clinker examined has shown C_5A_3; rarely can it be detected in commercial clinker, and then, in the experience of this laboratory, it has appeared only in traces in (questionably) the peculiar pleochroic unstable form. Rather, the disparity seems to reflect a disturbance of the phase relations indicated in the pure oxide quaternary diagram, probably through the universal presence of extraneous or accessory constituents such as magnesia, SO_3, alkalies, etc.

5. Finally, through the criteria developed in a study of experimental pure oxide mixes it becomes evident that most commercial clinkers contain lesser amounts of C_3A and C_4AF than are shown by calculation from chemical composition. Some clinkers, showing excellent development of C_4AF mass birefringence, attain nearly complete equilibrium crystallization; as a general rule, this structure is poorly developed, and in many cases there is a nearly complete absence of evidence of differentiation in the liquid phase. With no differentiation whatever, glass becomes the interstitial or matrix material in the clinker; with complete equilibrium differentiation, crystalline C_3A is the interstitial material; with partial differentiation, the case in most commercial clinker, C_3A and glass occur interstitially. The real problem of C_3A visibility thus becomes one of distinction between C_3A and glass. The work of this laboratory has provided some interesting information concerning the character of the glass phase; these observations, supplemented by further investigation, will appear in a later report.

Acknowledgments:

The author takes especial pleasure in acknowledging the whole-hearted assistance of Mr. M. A. Swayze of the Lone Star Cement Corp. in the preparation and analysis of the experimental burns, and expresses appreciation to the executives of the corporation for permission to publish this paper.

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SOME TESTS TO SHOW THE EFFECT OF FREEZING ON THE PERMEABILITY, STRENGTH, AND ELASTICITY OF CONCRETES AND MORTARS

BY HERBERT H. SCOFIELD[1]

SYNOPSIS

Some 1500 test specimens have been made and tested under laboratory conditions to show the effect of immediate freezing on some of the properties of concretes and mortars.

Six different types of cements were used in concretes or mortars with sand and gravel as aggregates.

Specimens to be frozen immediately were subjected, as soon as molded, to a freezing temperature of about 10 F. Freezing periods of 1 and 7 days were used.

Some specimens were subjected to a delayed freezing cycle in which the concrete was allowed to harden for various periods before freezing. The freezing period for all of these was 2 days at about 5 F.

The average loss in strength for concretes immediately frozen ran from about 40 to 60 per cent of the normal unfrozen strength at the same age. There was no systematic difference between the 1- and the 7-day freezing period, nor between rich and lean concretes. Dry concretes and those tested at 1 and 4 yr. seemed to show less proportionate percentage loss.

The permeability tests showed a large increase in leakage for the frozen specimens especially for those frozen 7 days. This was however proportionately much less for 1-yr. tests than for 7-day tests.

If the specimen was cured 24 to 47 hr. in warm dry air before freezing, practically no injury was done to the concrete. Cooler moist air precuring necessitated somewhat longer precuring before freezing in order not to injure the specimens.

Two-inch mortar cubes with or without hydrated lime addition suffered less percentage loss due to immediate freezing than did the concretes. Dry mortars were less injured than wet mortars.

The tests here reported were undertaken to throw some light on the question as to the amount of injury mortars or concretes sustain when frozen before or during the setting period. An actual case of frozen mortar on a stone masonry job furnishes the reason for the investigation.

At about the time these tests were started, Russian engineers reported the then prevailing practice of freezing concrete solid in the forms in the cold months followed by removal of the forms after the concrete had thawed and hardened in the spring. They reported no injury to the concrete.

MATERIALS

The materials used in these tests were bought on the local market. The aggre-

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gates were the usual central New York glacial drift gravels and sand. The sand was a washed sand showing not more than 2 per cent by weight silt content. The unit weights and sieve analyses of the aggregates are shown in Table I.

The cements used were as follows: cement No. 1 was an ordinary portland cement; No. 2, a high-early-strength portland cement; No. 3, a water-proofed portland cement; No. 4, a high-alumina cement; Nos. 5 and 6 were masonry cements.

TABLE I.—CHARACTERISTICS OF AGGREGATES USED.

Aggregates measured room-dry loose.

The fine and coarse aggregates were combined in such proportions as to give approximately straight-line variation of sizes.

Aggregate	Weight, lb. per cu. ft.	Per cent by Weight											
		Passing a 1½-in. Sieve	Passing a 1¼-in. Sieve	Passing a 1-in. Sieve	Passing a ¾-in. Sieve	Passing a ½-in. Sieve	Passing a ¼-in. Sieve	Passing a No. 4 Sieve	Passing a No. 8 Sieve	Passing a No. 14 Sieve	Passing a No. 28 Sieve	Passing a No. 48 Sieve	Passing a No. 100 Sieve
Coarse	88.0	88.7	53.2	13.5	1.7								
Medium	92.7			97.5	65.6	40.4	9.65						
Fine	102.5							100.0	89.4	70.4	50.8	12.3	2.6

TABLE II.—CHARACTERISTICS OF CEMENTS USED.

Results from tests of concretes using cements Nos. *2a* and *2b* were averaged together and designated as cement No. 2.

	CEMENT NO. 1	CEMENT NO. *2a*	CEMENT NO. *2b*	CEMENT NO. 3	CEMENT NO. 4
Normal consistency	24	28	27	26.0	23.0
Soundness	O.K.	O.K.	O.K.	O.K.	O.K.
Initial set (Gilmore)	2 hr. 45 min.	3 hr. 20 min.	3 hr. 25 min.	3 hr. 30 min.	3 hr. 40 min.
Final set (Gilmore)	7 hr. 30 min.	6 hr. 0 min.	9 hr. 0 min.	8 hr. 0 min.	7 hr. 30 min.
Retained on No. 200 sieve, per cent	10.5	2.0	4.0	11.1	7.0
1 to 3 standard briquets					
1 day	...	191	233	...	387
3 days	...	330	360	...	408
7 days	327	400	432	255	410
28 days	383	495	447	365	412

The hydrated lime used in some of the mortar tests was an ordinary brand used in masonry. The characteristics of the cements are shown in Table II.

TEST METHODS

The concrete in these tests was machine mixed. The mortars were hand and trowel mixed in pans.

Concrete strength tests were made on standard 6 by 12-in. cylinders. Deformations were determined on a 10-in. gage length with a dial compressometer reading directly to 0.0005 in.

Permeability specimens 4 in. in thickness and 12 in. in diameter were molded from the corresponding strength-test batches and subjected to the same treatment. Permeability was measured by an apparatus which subjected a 6-in. circular space of the original bottom of the specimen to water under 25 lb. per sq. in. pressure. The amount of water entering or passing through the specimen was determined.

Mortar strengths were determined on 2-in. cubes tested on sides without capping.

Freezing procedure is best shown by the temperature diagrams, Fig. 1. The test ages given are ages exclusive of freezing period.

TEST DATA

The test data are shown in Tables III to VIII inclusive and average results illustrated in Figs. 1 to 3. The results given are the average of at least three batches made on different days. Test specimens were distributed over time intervals in such a way as to give best dispersion of results.

DISCUSSION OF DATA

For the concretes and mortars used and under the conditions prevailing in these tests, the following seems to be indicated by the results obtained:

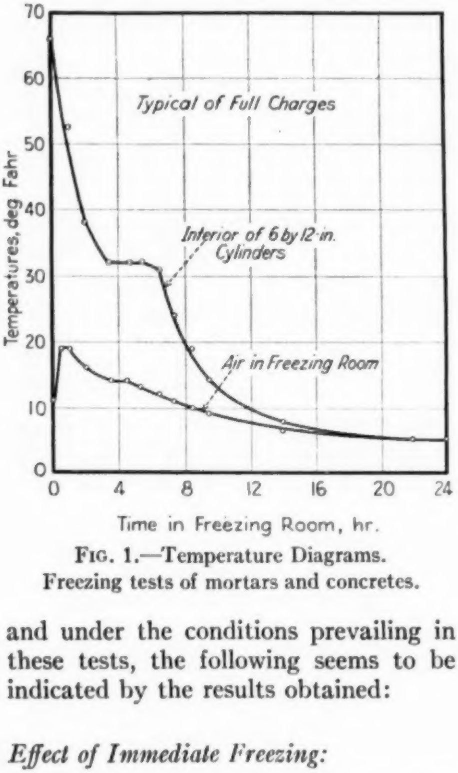

FIG. 1.—Temperature Diagrams.
Freezing tests of mortars and concretes.

Effect of Immediate Freezing:

1. Concrete frozen immediately after placing attains on the average about 50 per cent of the strength of the normal unfrozen concrete at the same age. There is little difference in this respect between a 1-day and a 7-day freezing period.

2. There is some indication that older concretes have less proportionate loss of strength due to immediate freezing than those tested at earlier periods.

3. There is some indication that dry concretes suffer less injury than wet concretes, especially at early ages.

4. There is little difference between a rich mix and a lean mix in their resistance to immediate freezing.

5. The high-alumina cement was the only cement to show indications of setting and hardening during the freezing period.

6. The rate of leakage of water in the permeability tests may be taken as a measure of the relative porosity of the concretes. Concretes which had little porosity unfrozen showed relatively great porosity after being frozen before hardening.

7. There seems to be a great deal more leakage for concretes frozen for 7 days than for 1 day.

8. The leakage for frozen specimens tested at 1 yr. was much less than when tested at 7 days.

Effect of Delayed Freezing:

1. The results seem to indicate that dry concretes (2-in. slump) may be frozen solid without practical injury if cured for 24 to 48 hr. in room-dry conditions before freezing.

2. Wet concretes (9-in. slump) require considerably longer time of precuring before freezing in order to be uninjured by freezing.

3. Moist-cured specimens require a longer period of precuring than dry-cured specimens before freezing in order that the concrete shall be uninjured by freezing.

Effect of Immediate Freezing on Strength of Mortars:

1. The 2-in. cube mortar tests indicate less percentage loss due to freezing than do the concrete tests.

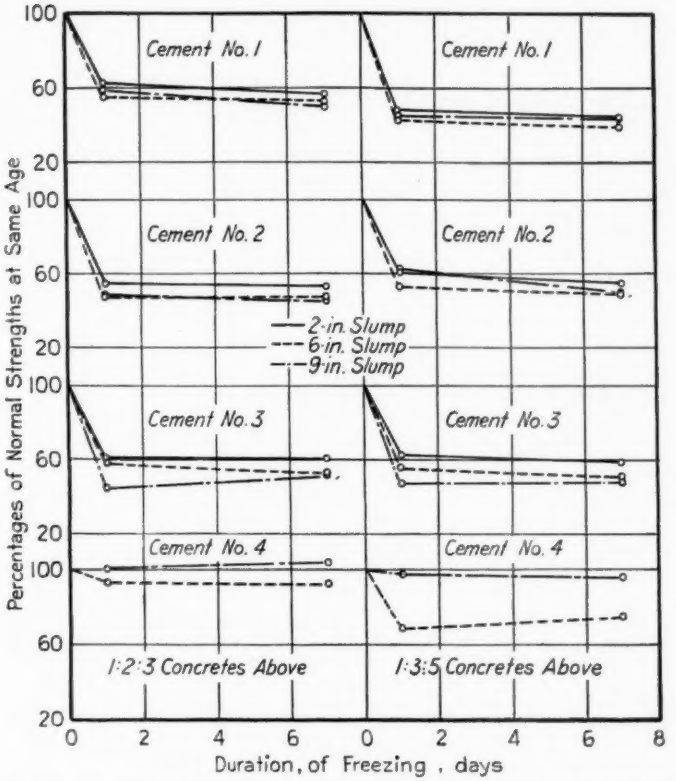

FIG. 2.—Effect of Immediate Freezing on Strength of Concrete. Averages for all ages.

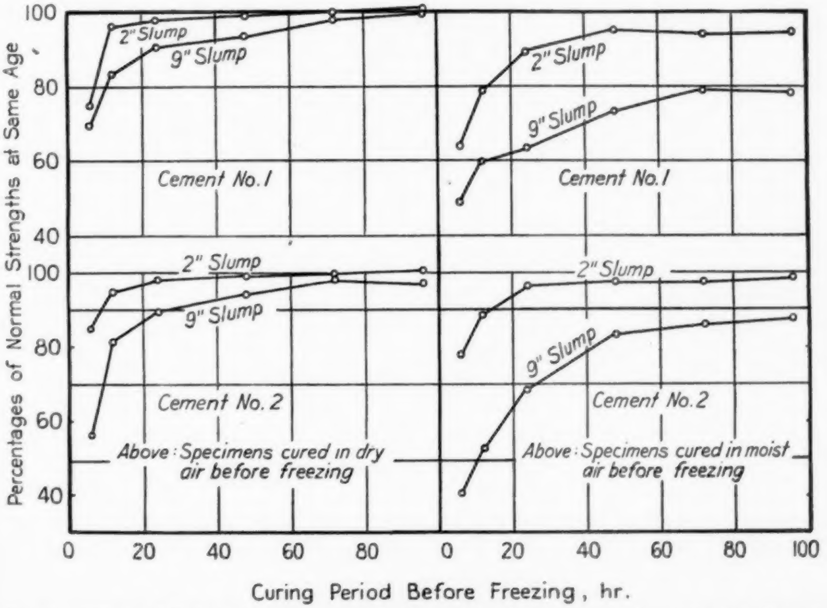

FIG. 3—Effect of Delayed Freezing on the Strength of Concrete. Averages for all ages.

2. Dry mortars were injured less than wet mortars.

3. The addition of 10 per cent hydrated lime did not appreciably affect the results as to the effect of freezing.

TABLE III.—EFFECT OF IMMEDIATE FREEZING ON THE COMPRESSIVE STRENGTH OF CONCRETE.

Actual compressive strength in pounds per square inch is given for all normal (0-days frozen) results.
Ages given are for moist curing periods after frozen period.

Cement	Slump, in.	Frozen Period, days	Compressive Strength of Previously Frozen Concrete as Percentage of the Strength of the Normal Concrete at the Same Age									
			1:2:3 Concrete					1:3:5 Concrete				
			7 days	28 days	1 yr.	4 yr.	Average, per cent	7 days	28 days	1 yr.	4 yr.	Average, per cent
No. 1...........	2	0	2387	4180	6773	6787	100	1502	2777	4045	4665	100
		1	61.6	55.1	64.4	67.8	62.2	40.2	43.2	55.5	55.4	48.6
		7	57.6	51.0	55.6	64.3	57.1	38.5	37.4	53.4	51.7	45.2
No. 2...........	2	0	4000	4996	6370	7040	100	3075	3690	4815	4596	100
		1	50.0	61.7	57.0	58.4	54.8	54.0	67.7	63.1	71.8	60.6
		7	44.2	56.7	60.5	53.8	53.8	42.3	56.9	59.4	58.7	54.3
No. 3...........	2	0	1750	3055	4335	4567	100	1090	1990	3760	3061	100
		1	44.6	48.5	73.4	77.2	60.9	51.4	50.2	70.3	76.0	62.3
		7	52.0	51.7	72.2	65.7	60.4	50.4	49.2	60.0	77.7	58.7
No. 1...........	6	0	2300	3677	6046	5685	100	1118	2033	3433	4063	100
		1	48.8	51.2	55.1	67.7	55.7	37.2	37.0	44.4	50.6	42.8
		7	47.5	50.1	54.7	63.1	53.8	33.7	37.2	43.7	45.1	39.9
No. 2...........	6	0	3605	4630	5572	5692	100	2390	3290	3990	3656	100
		1	38.4	46.0	51.3	55.2	47.7	50.6	57.1	50.9	57.1	53.9
		7	36.9	43.5	52.8	56.3	47.4	43.0	43.4	50.0	61.0	49.3
No. 3...........	6	0	1750	3160	4725	5291	100	990	1770	3250	3005	100
		1	45.7	57.9	70.9	65.3	58.7	51.1	57.7	52.9	59.9	55.4
		7	46.8	49.7	55.7	60.0	53.5	48.0	50.9	50.6	57.9	51.8
No. 4...........	6	0	4970	4922	5755	5872	100	2775	2780	3538	2788	100
		1	87.7	97.7	88.5	101.0	93.7	69.0	70.0	65.4	68.5	68.2
		7	85.0	97.0	97.4	92.7	93.0	70.3	77.3	81.0	72.7	75.3
No. 1...........	9	0	1840	3047	5123	4738	100	825	1453	2640	2950	100
		1	53.0	54.7	60.5	71.1	60.6	49.0	49.3	52.1	62.6	53.2
		7	38.7	52.0	51.3	64.7	51.7	39.7	46.0	46.8	54.4	46.7
No. 2...........	9	0	3100	4085	5230	4976	100	1765	2300	3290	2300	100
		1	41.4	46.2	51.4	57.0	49.0	57.8	57.9	66.9	68.6	62.8
		7	38.7	45.4	49.8	55.2	47.3	39.7	53.5	44.1	64.9	50.5
No. 3...........	9	0	1000	1600	4100	4272	100	600	945	2550	2520	100
		1	41.0	44.7	45.4	48.4	44.9	40.4	50.7	43.6	52.1	46.9
		7	41.0	55.0	49.8	63.8	52.6	44.2	57.2	42.7	47.8	47.9
No. 4...........	9	0	4253	4545	5283	4497	100	2858	2940	3277	2700	100
		1	103.8	101.5	91.5	106.1	100.7	91.5	94.2	106.8	103.0	98.9
		7	106.0	104.0	105.2	105.0	105.0	93.8	89.4	100.9	103.3	96.7

CONCLUSIONS

It is of course understood that concrete and mortar subjected to freezing temperature on the job may or may not be frozen. Many factors govern.

It is true also that a difference in the effect of freezing may exist between a small mass such as a 6 by 12-in. cylinder or a 2-in. cube and the larger mass of the job concrete.

For concrete and mortar which actually do freeze after being placed, it is felt that percentage losses in the tests reported are applicable and significant.

Acknowledgments:

Acknowledgment is made to Messrs. E. C. MacCubbin, J. W. Rowe, A. W. Young, F. E. Olditch, G. P. Church, and J. M. Walsh for helping in these tests, and to Mr. A. N. Vanderlip, Instructor in Materials, for advice and help in the performance of the investigations.

TABLE IV.—EFFECT OF IMMEDIATE FREEZING ON THE MODULUS OF ELASTICITY OF CONCRETE.

Actual modulus in million pounds per square inch units is given for all normal (0-days frozen) results.
All moduli computed at stress of 0.4 the ultimate of normal at same age.
Ages given are moist curing after frozen period.

Cement	Slump, in.	Frozen Period, days	Modulus of Elasticity of Previously Frozen Concrete as Percentage of the Modulus of the Normal at the Same Age									
			1:2:3 Concrete					1:3:5 Concrete				
			7 days	28 days	1 yr.	4 yr.	Average, per cent	7 days	28 days	1 yr.	4 yr.	Average, per cent
No. 1	2	0	2.17	3.16	3.67	4.31	100.0	2.00	2.52	3.60	3.12	100.0
		1	62.6	63.8	87.0	86.5	75.0	20.0	41.7	25.5	66.7	38.5
		7	37.4	63.8	58.5	79.3	59.7	....	....	44.2	63.8	....
No. 2	2	0	2.44	2.93	3.25	3.80	100.0	2.32	2.69	3.22	3.17	100.0
		1	59.8	78.6	83.3	74.7	74.1	44.8	88.6	80.0	76.5	72.0
		7	43.3	71.0	59.0	59.5	58.2	44.8	72.4	67.2	67.5	63.0
No. 3	2	0	2.06	2.54	2.79	3.46	100.0	1.89	2.16	2.83	3.05	100.0
		1	35.8	72.8	91.2	84.2	71.0	26.1	35.9	63.8	88.8	53.6
		7	50.8	80.0	91.2	91.5	78.4	20.9	61.7	84.0	80.0	61.6
No. 1	6	0	2.13	3.88	3.48	3.98	100.0	2.04	2.38	2.85	2.81	100.0
		1	54.4	51.3	82.5	95.0	70.8	....	....	49.1	57.0	....
		7	44.5	31.2	68.6	89.1	58.3	....	....	45.3	49.1	....
No. 2	6	0	2.27	2.81	3.81	3.67	100.0	2.17	2.54	3.20	3.02	100.0
		1	14.4	49.2	66.2	76.2	51.5	62.9	74.3	68.3	65.7	67.8
		7	....	40.8	58.5	73.6	45.7	50.0	....	49.0	65.7	....
No. 3	6	0	2.18	2.29	3.10	3.52	100.0	1.89	2.15	2.28	3.08	100.0
		1	58.3	91.6	87.2	98.0	90.3	65.7	90.0	81.5	98.0	81.9
		7	....	....	....	31.5	....	26.9	33.0	50.5	86.5	37.7
No. 4	6	0	3.00	3.42	3.60	3.86	100.0	2.11	2.29	2.45	2.36	100.0
		1	88.0	84.0	96.7	98.3	92.9	68.8	46.0	69.0	80.0	65.9
		7	91.6	81.0	87.5	96.3	91.5	74.5	76.4	78.5	86.0	78.8
No. 1	9	0	2.23	2.49	3.60	3.70	100.0	1.50	2.23	2.62	2.36	100.0
		1	47.5	48.2	85.0	96.4	69.3	18.0	30.0	51.5	55.1	38.6
		7	....	36.2	47.5	86.5	....	....	28.0	33.2	50.0	....
No. 2	9	0	2.35	2.44	3.13	3.23	100.0	2.06	1.84	2.64	2.96	100.0
		1	26.5	62.4	67.0	81.4	59.3	81.0	94.0	73.5	59.6	77.0
		7	14.7	65.2	64.4	79.8	56.0	31.0	55.6	45.5	51.7	45.9
No. 3	9	0	1.60	2.21	2.28	3.23	100.0	1.33	2.00	1.96	2.89	100.0
		1	19.5	44.0	77.0	53.0	48.4	13.8	68.0	40.0	54.7	44.1
		7	12.5	64.5	65.8	75.8	54.6	25.0	50.0	40.0	42.7	39.4
No. 4	9	0	2.70	2.98	3.30	2.95	100.0	2.37	2.93	2.47	2.58	100.0
		1	81.8	97.2	84.4	94.0	90.0	100.0	90.0	95.0	95.5	95.1
		7	96.6	99.5	98.5	95.4	99.0	100.0	85.0	98.5	92.5	94.0

TABLE V.—EFFECT OF DELAYED FREEZING ON THE COMPRESSIVE STRENGTH OF CONCRETE.
All specimens cured in moist air at approximately 70 F. after freezing and thawing.

Cement	Water-Cement Ratio, gal. per sack	Slump, in.	Curing Period Before Freezing, hr.	Compressive Strength, lb. per sq. in. Concrete Previously Frozen Expressed as Percentage of Normal Strength at Same Age. Freezing Period in all Cases 48 hr. at 5 F.							
				Cured in Dry Air Before Freezing				Cured in Moist Air Before Freezing			
				7 days	28 days	1 yr.	Average, per cent	7 days	28 days	1 yr.	Average, per cent
No. 1	8.5	2	Normal	1810	2935	4400	100.0	1810	2935	4400	100.0
			6	72.0	74.0	78.1	74.7	58.5	62.5	71.0	64.0
			12	93.0	97.4	98.0	96.1	75.0	80.0	81.0	78.7
			24	98.0	99.0	98.0	98.0	85.0	93.6	89.0	89.2
			48	99.0	99.5	98.5	99.0	88.5	94.0	103.0	95.2
			72	100.0	100.0	99.0	100.0	87.3	97.6	99.0	94.6
			96	103.0	102.0	99.0	101.0	87.3	95.8	101.0	95.0
	11.75	9	Normal	923	1597	3025	100.0	923	1597	3025	100.0
			6	58.3	77.0	73.8	69.7	45.5	51.2	50.0	48.9
			12	74.6	89.0	87.0	83.5	51.5	62.0	66.0	59.8
			24	86.6	90.5	95.0	90.7	48.8	69.0	72.0	63.3
			48	90.0	98.0	92.4	93.5	54.0	80.0	85.8	73.3
			72	101.0	96.0	97.0	98.0	66.5	85.0	86.0	79.2
			96	101.0	98.0	100.0	99.6	69.5	81.4	84.5	78.5
No. 2	8.5	2	Normal	3450	4206	5022	100.0	3450	4206	5022	100.0
			6	78.0	88.5	88.5	85.0	83.2	74.5	76.0	77.9
			12	92.5	100.0	92.0	94.8	96.2	83.5	86.0	88.6
			24	95.0	103.0	96.0	98.0	99.0	95.5	94.0	96.2
			48	97.0	100.0	100.0	99.0	98.5	96.5	98.0	97.7
			72	97.0	104.0	98.0	99.6	100.5	93.5	99.0	97.6
			96	98.5	103.0	99.5	100.3	100.7	95.5	98.0	98.1
	12.25	9	Normal	1673	2400	3190	100.0	1673	2400	3190	100.0
			6	49.4	60.5	58.4	56.1	37.0	41.4	43.7	40.7
			12	69.5	86.0	88.0	81.2	45.8	56.0	57.0	52.9
			24	86.2	92.0	90.0	89.4	60.6	72.0	73.0	68.5
			48	96.5	92.5	94.5	94.5	79.0	86.3	85.5	83.6
			72	102.0	96.0	96.0	98.0	82.0	86.1	90.0	86.0
			96	98.0	98.0	95.0	97.0	80.0	89.5	93.0	87.5

TABLE VI.—EFFECT OF IMMEDIATE FREEZING ON THE COMPRESSIVE STRENGTH OF 1 TO 1 MORTARS.

Ages given are for moist curing period after freezing period.
Hydrated lime added was 10 per cent of weight of cement.

Cement	Consistency	Frozen Period, days	Compressive Strength, lb. per sq. in. Mortar Previously Frozen Expressed as Percentage of Normal Strength at Same Age							
			Without Hydrated Lime				With Hydrated Lime			
			7 days	28 days	1 yr.	Average, per cent	7 days	28 days	1 yr.	Average, per cent
No. 1	Dry	0	3405	3738	5789	100.0	4014	5757	7191	100
		1	80.7	96.7	80.5	86.0	90.0	81.5	91.9	87.8
		7	71.0	80.4	86.6	79.3	69.6	71.3	85.2	75.4
	Wet	0	5238	7096	10234	100.0	3906	5418	8864	100.0
		1	71.4	77.6	80.2	76.4	78.2	81.4	85.1	81.6
		7	59.1	61.1	78.1	66.1	58.2	72.4	74.8	68.5
No. 2	Dry	0	5518	5770	6489	100.0	5533	6450	7785	100.0
		1	69.4	91.1	93.3	84.6	82.6	89.0	89.1	86.9
		7	59.3	66.4	85.2	70.3	70.3	76.7	84.0	77.0
	Wet	0	7116	8198	10699	100.0	5717	6680	8859	100.0
		1	77.8	83.3	75.5	78.8	75.0	84.6	76.5	78.9
		7	57.0	64.6	63.1	61.6	59.6	62.0	69.1	63.6
No. 3	Dry	0	2300	2961	3965	100.0	2721	3538	5242	100.0
		1	99.5	88.6	97.6	95.2	90.7	90.2	88.3	89.7
		7	93.4	85.1	97.6	92.0	88.5	80.9	79.0	82.8
	Wet	0	3816	5313	7406	100.0	2778	4501	6095	100.0
		1	83.7	88.0	101.5	91.1	93.7	79.5	90.6	87.9
		7	84.6	75.7	87.6	81.0	97.0	73.0	98.2	89.4
No. 5	Dry	0	447	1025	2098	100.0	....	....	....	....
		1	67.0	67.7	62.5	65.7	....	....	....	....
		7	80.3	87.6	63.0	76.9	....	....	....	....
	Wet	0	301	818	1644	100.0	....	....	....	....
		1	79.7	67.0	84.9	77.2	....	....	....	....
		7	96.6	70.0	85.8	84.1	....	....	....	....
No. 6	Dry	0	2190	3182	5048	100.0	....	....	....	....
		1	75.2	91.2	90.0	85.5	....	....	....	....
		7	77.8	77.1	84.6	79.8	....	....	....	....
	Wet	0	1071	1812	3127	100.0	....	....	....	....
		1	87.9	89.2	103.0	93.4	....	....	....	....
		7	91.6	83.0	97.7	90.8	....	....	....	....

TABLE VII.—EFFECT OF IMMEDIATE FREEZING ON THE COMPRESSIVE STRENGTH OF 1 TO 3 MORTARS.

Ages given are for moist curing period after freezing period.
Hydrated lime added was 10 per cent of weight of cement.

Cement	Consistency	Frozen Period, days	Compressive Strength, lb. per sq. in. Mortar Previously Frozen Expressed as Percentage of Normal Strength at Same Age							
			Without Hydrated Lime				With Hydrated Lime			
			7 days	28 days	1 yr.	Average, per cent	7 days	28 days	1 yr.	Average, per cent
No. 1	Dry	0	1548	2232	4117	100.0	1478	1950	3264	100.0
		1	77.6	89.5	76.0	81.0	83.2	92.7	91.5	89.1
		7	82.4	92.6	75.8	83.6	84.6	82.2	91.8	86.2
	Wet	0	2218	3566	5476	100.0	1887	2846	4873	100.0
		1	60.9	82.8	91.4	78.3	71.8	89.7	99.0	86.8
		7	68.3	82.0	96.7	82.3	75.5	93.8	98.5	89.3
No. 2	Dry	0	3230	3704	5063	100.0	3088	3458	4552	100.0
		1	78.2	94.4	87.3	86.6	82.5	86.9	102.0	90.5
		7	77.4	91.6	86.2	85.1	78.0	86.5	84.2	82.9
	Wet	0	3842	4842	6403	100.0	4108	4654	6257	100.0
		1	88.2	94.0	102.0	94.7	81.6	91.3	92.5	88.5
		7	73.4	86.8	96.5	85.6	63.4	84.5	82.0	76.5
No. 3	Dry	0	2284	3041	4443	100.0	2098	3192	4055	100.0
		1	86.0	94.2	96.9	92.3	94.1	86.7	105.0	95.3
		7	80.5	87.6	86.1	84.7	90.0	78.5	96.9	88.5
	Wet	0	1569	2156	3841	100.0	1687	2608	3979	100.0
		1	86.9	98.1	102.3	95.8	94.0	91.6	105.4	97.0
		7	82.5	98.6	102.8	94.6	80.3	88.0	86.2	84.8
No. 5	Dry	0	292	685	1278	100.0	....	....	....	....
		1	74.0	80.6	101.8	85.5	....	....	....	....
		7	87.7	82.9	116.8	95.8	....	....	....	....
	Wet	0	88	262	389	100.0	....	....	....	....
		1	76.1	66.0	122.1	88.1	....	....	....	....
		7	94.2	66.0	132.0	97.4	....	....	....	....
No. 6	Dry	0	768	1083	1958	100.0	....	....	....	....
		1	76.0	92.3	102.3	90.2	....	....	....	....
		7	71.5	98.6	109.2	93.1	....	....	....	....
	Wet	0	321	533	768	100.0	....	....	....	....
		1	87.8	84.8	107.8	93.5	....	....	....	....
		7	90.5	90.4	129.3	103.4	....	....	....	....

TABLE VIII.—EFFECT OF IMMEDIATE FREEZING ON THE PERMEABILITY OF CONCRETE.

These batches were taken from the corresponding strength test batches.
Ages given are for moist curing periods following frozen period.

Cement	Slump, in.	Frozen Period, days	Leakage at 25 lb. per sq. in. Pressure, gal. per sq. ft. per hr.			
			1:2:3 Concrete		1:3:5 Concrete	
			7 days	1 yr.	7 days	1 yr.
No. 1	2	0	....	....	0.014	0.004
		1	....	....	0.860	0.040
		7	....	....	1.690	1.320
	6	0	....	....	0.007	0.006
		1	....	....	1.800	0.071
		7	....	....	20.000	1.700
	9	0	....	....	0.049	0.002
		1	....	....	4.500	1.150
		7	....	....	30.000	2.480
No. 2	2	0	0.001	0.001	0.012	0.006
		1	0.015	0.004	0.670	0.125
		7	1.353	0.005	6.300	0.172
	6	0	0.000	0.000	0.007	0.008
		1	0.566	0.096	3.090	0.517
		7	3.055	0.130	18.550	2.500
	9	0	0.015	0.007	0.112	0.050
		1	0.740	0.217	12.400	1.200
		7	18.600	1.350	22.400	5.200
No. 3	2	0	0.008	0.036	0.009	0.012
		1	0.443	0.070	1.280	0.153
		7	1.555	0.155	8.700	0.700
	6	0	0.009	0.058	0.020	0.000
		1	0.440	0.400	3.540	0.240
		7	15.500	1.230	15.500	2.325
	9	0	0.001	0.000	0.040	0.030
		1	3.450	2.520	2.30	0.780
		7	18.600	3.750	24.800	4.477
No. 4	No leakage normal or frozen.					

DISCUSSION

Mr. C. H. Scholer.[1]—Mr. Scofield has made a valuable contribution to our knowledge of the effect of low temperature during the construction period upon the quality of the concrete produced. An engineer is prone to base his predictions as to the probable strength of the concrete in his structure upon data secured in the laboratory under normal temperature conditions. In actual procedure these conditions are seldom obtained.

I gather from Mr. Scofield's paper that after the various freezing periods the test specimens were again subjected to normal curing conditions. It would be interesting to know of the effect of long-continued low temperatures, not much above the freezing point. This condition is very frequently met with in practice and may have a very detrimental effect upon the resulting concrete.

I was particularly interested in the author's observation that mortars were less severely damaged than were concretes containing coarse aggregate. I have observed this condition in construction and have attributed it to the accumulation of water under the pieces of coarse aggregate due to bleeding.

It is likely that the injurious effect of freezing and low-temperature curing upon durability and resistance to freezing and thawing in the finished structure is even more pronounced than its effect upon compressive strength and permeability.

Mr. H. H. Scofield[2] *(author's closure, by letter)*.—The effect of low temperatures of about 33 to 50 F. during the curing period has been shown by numerous investigators. The author's investigation attempts to cover the much less common condition—when the concrete or mortar is actually frozen hard during the curing period. The effect of the latter, of course, is a loss of strength due to the disruptive forces of the ice crystals formed. The porosity is increased and as Scholer points out, the durability and resistance to freezing and thawing would likely be greatly reduced.

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PLASTIC FLOW AND VOLUME CHANGES OF CONCRETE

By Raymond E. Davis,[1] Harmer E. Davis,[1] and Elwood H. Brown[1]

Synopsis

Recent findings of investigations carried on at the University of California on plastic flow of concrete are discussed.

In tests under long-time loading it was found that even after 10 yr. some plastic flow was still discernible in plain concretes under sustained stresses of several hundred pounds per square inch; however, over 95 per cent of the probable total flow took place within 5 yr. In a series of tests on reinforced concrete columns under load for more than 5 yr., it was found that, in terms of stresses in the steel or concrete, the movements due to drying shrinkage or to plastic flow are of practical importance principally during the first year.

Tests to determine the effect of water-cement ratio and of aggregate-cement ratio upon plastic flow indicated that the denser the cement paste, the less the flow and that this variable was very important. Within the range of normal concretes tested, it was observed that with pastes of the same quality, the flow was practically proportional to the paste content of the concrete.

In tests to compare the flow of concretes in axial tension and compression, it was observed that, at least during the early ages, the flow under tensile stress was greater than that under compressive stress. At the later ages, the rate of flow was less under tensile than under compressive stress. Similar results were found in tests to determine the fiber strains in plain concrete beams under constant sustained bending moment.

A low-heat type of portland cement was found to exhibit markedly greater plastic properties, particularly at the early ages, than a normal portland cement. This is discussed in relation to volume changes and cracking of concrete structures.

In tests made under mass-curing conditions, the stresses developed due to thermal changes in large concrete cylinders under complete axial restraint were measured. It was indicated that the degree to which flow takes place has an important influence upon the stresses developed.

In the technical literature of a decade ago, there was frequent reference to the strength and elasticity of concrete, but only an occasional reference to shrinkage due to drying and almost no reference to the gradual deformation under the action of sustained load, which has been variously called "creep," "time yield," and "plastic flow."

As a result of the research of recent years, there has been developed a general conception of the effect of shrinkage and plastic flow upon the behavior of con-

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crete structures, which is already leading to marked changes in design and construction practices.

It is now generally believed that shrinkage and plastic flow are closely related phenomena, each being primarily due to changes in the amount of adsorbed water in the cement gel and being but little directly influenced by the free water occupying the pore spaces within the concrete mass.

In spite of the rather extensive researches that have been made in this field, it is not yet possible quantitatively to state with any degree of certainty what is likely to be the magnitude of either plastic flow or shrinkage under the conditions which surround any given concrete structure, nor is it possible quantitatively to state with accuracy what is the effect of plastic flow and shrinkage upon the magnitude and distribution of stresses.

The property of shrinkage of concrete due to drying is altogether undesirable, but while probably shrinkage can never be entirely eliminated, yet there seems to be the possibility, through the proper selection of materials and methods, of reducing shrinkage to a point where it will not be a factor for serious consideration—certainly to a point far below that observed in present-day concretes. On the whole, plastic flow does not seem to be an undesirable property. In certain reinforced concrete members it tends to make possible more efficient use of steel, and in thin structures subjected to drying, as well as in mass structures subjected to thermal changes due to the hydration of cement, it tends to promote a more favorable distribution of stresses than would otherwise exist.

The purpose of this paper is to present the results of some of the investigations made at the University of California, all having to do with volume changes of concrete, and to point out the possible significance of the findings as applied to design and construction problems. By reason of space limitations, the paper will be devoted principally to a consideration of volume changes under the action of sustained load, hereinafter referred to as *plastic flow*.

SCOPE OF INVESTIGATIONS

Started in 1926, plastic flow investigations at the Engineering Materials Laboratory, University of California, have comprised to date some 25 series of tests. Among the earlier series were included studies to determine the effect upon plastic flow under sustained compressive stress of (1) duration of loading, (2) moisture and temperature conditions of storage, (3) moisture content of concrete, (4) gradation and mineral character of the aggregate, (5) size of mass, (6) age at time of load application, (7) intensity of sustained stress, and (8) reinforcement. Previous reports[2] have described and discussed the results of the tests having to do with these variables.

Recent series of tests, undertaken during the period 1934 to 1937, have included studies to determine (1) effect of water-cement ratio and of aggregate-cement ratio upon plastic flow, (2) effect of fineness and composition of cement upon plastic flow, (3) plastic flow of concretes in tension and in compression, (4) fiber strains in plain concrete beams under constant sustained bending moment, and (5) stresses developed in large concrete cylinders under complete axial restraint during a heating-cooling cycle similar to that which will occur in mass concrete.

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[2] Raymond E. Davis, "Flow of Concrete Under Sustained Compressive Stress," *Proceedings*, Am. Concrete Inst., Vol. 24, p. 303 (1928).

R. E. Davis and H. E. Davis, "Flow of Concrete Under Sustained Compressive Stress," *Proceedings*, Am. Soc. Testing Mats., Vol. 30, Part II, p. 707 (1930).

Raymond E. Davis and Harmer E. Davis, "Flow of Concrete Under the Action of Sustained Loads," *Proceedings*, Am. Concrete Inst., Vol. 27, p. 837 (1931).

R. E. Davis, H. E. Davis and J. S. Hamilton, "Plastic Flow of Concrete Under Sustained Stress," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 354 (1934).

The following conditions have prevailed throughout all the tests. For specimens maintained at constant temperature, the temperature has been controlled to within 1 deg. Fahr. of the nominal temperature. For specimens exposed to air, the humidity has been generally maintained within 1 per cent relative humidity of the nominal value. Measurements of surface changes in length have been made with fulcrum-plate strain gages, equipped with micrometer dial gages reading to 0.00001 in. by estimation. Measurements of interior changes in length have been made with Carlson electric strainmeters.[3] Beam deflections have been measured with specially constructed deflectometers, the indicating elements of which are micrometer dial gages, reading to 0.00001 in. by estimation. Axial compressive and tensile loads have been maintained through the use of heavy coil springs.

For most of the series of investigations, a test group has included three similar specimens under load and at least two without load, for the purpose of measuring changes due to causes other than load. There are at present under test in the laboratory more than 500 specimens.

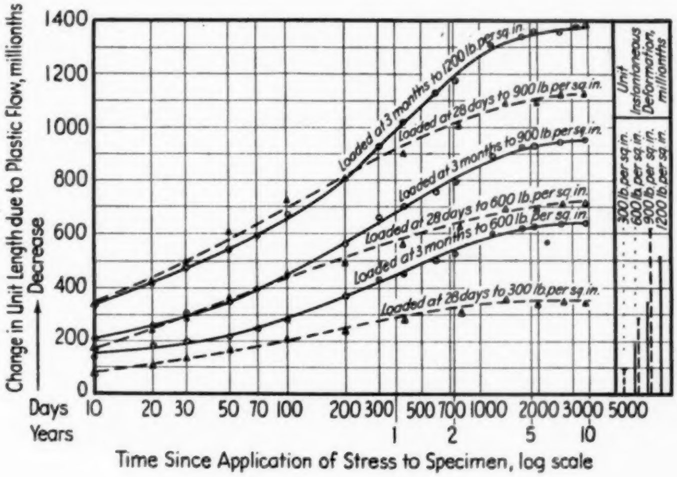

FIG. 1.—Flow Under Long-Sustained Stress.

Flow tests, series No. 2. Specimens, 4 by 14-in. cylinders. Aggregate, 0 to 1½-in. crushed granite. Fineness modulus = 5.03. Cement, normal portland. Aggregate-cement ratio by weight = 5.05. Water-cement ratio by weight = 0.69. Slump = 3 in. Curing, moist at 70 F. (21 C.) until time of loading. Age at loading, as indicated. Sustained stress, compression, as indicated. Storage after loading, in air at 70 F. (21 C.) and 70 per cent relative humidity.

RESULTS OF INVESTIGATIONS

Plastic Flow Under Long-Time Loading:

The plastic flows of plain concrete cylinders which have been under load for 10 yr. are shown in Fig. 1. One group of specimens was loaded at the age of 28 days, and the other at the age of 3 months; prior to loading, the concrete was moist cured, and after loading, it was stored in air at 70 per cent relative humidity and 70 F. The sustained compressive stresses were 300, 600, 900, and 1200 lb. per sq. in.

Although under the sustained stress of

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[3] R. E. Davis and R. W. Carlson, "The Electric Strain Meter and Its Use in Measuring Internal Strains," *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part II, p. 793 (1932).
R. W. Carlson, "Five Years' Improvement of the Elastic Wire Strain Meter," *Engineering News-Record*, Vol. 114, May 16, 1935, p. 696.

300 lb. per sq. in., flow apparently ceased after 3 or 4 yr. under load, at the higher stresses, measurable although small changes in length due to flow appear still to be taking place. For the concrete loaded at the age of 28 days to a stress of 900 lb. per sq. in., the flow after 10 yr. under load has amounted to 1130 millionths per unit, or more than 1.3 in. per 100 ft. About 80 per cent of the total flow took place within the first year under load and about 97 per cent within 5 yr.

An approximate proportionality of stress to deformation appears to have been maintained over the loading period. For the same sustained stress, the age at loading appears not to have a large effect upon the rate of change in length after the first few months under load.

Results comparable with those shown in Fig. 1 were obtained from tests upon similar groups of plain concrete specimens which were stored under water for about 7 yr. Although for the water-stored specimens at the early ages the rate of flow was much less and at the end of 7 yr. the magnitudes of the deformations due to sustained load were of the order of one third to one half those for the air-stored specimens, the *rate* of flow at the later ages was somewhat greater for the water-stored specimens.

In Table I are summarized the results of tests upon reinforced concrete columns under load for about 5½ yr. There are shown the stresses in the longitudinal steel and in the concrete due to the combined effect of elastic and plastic strains produced by the sustained load and of length changes due to causes other than applied load. As flow progressed, more and more load was transferred from the concrete to the steel, and the rate of flow decreased in much greater proportion than would be the case for plain concrete sustaining the same initial stress. Although a detectable movement appears to continue for a long time, in terms of stress in the steel reinforcing, the changes in length appear to be unimportant after the age of a year or so.

It will be noted that while under conditions of wet storage the stress in the

TABLE I.—UNIT STRESSES IN REINFORCED-CONCRETE COLUMNS.

Nominal Strength of Concrete at 28 Days, lb. per sq. in.	Steel Ratio, per cent	Total Load Applied to Column, lb.	Unit Stresses, lb. per sq. in.							
			At Time of Application of Load		1 yr. Under Load		3 yr. Under Load		5½ yr. Under Load	
			Steel	Concrete	Steel	Concrete	Steel	Concrete	Steel	Concrete
COLUMNS STORED IN AIR OF 50 PER CENT RELATIVE HUMIDITY AT 70 F.										
2000.......	5.0	22 300	9660	875	26 900	−20[a]	27 400	−50[a]	28 000	−75[a]
2000.......	1.9	14 200	6540	610	34 800	60	35 700	45	37 100	15
4000.......	1.9	21 800	7860	975	37 500	395	40 400	340	41 700	315
COLUMNS STORED UNDER WATER AT 70 F.										
2000.......	5.0	19 200	7200	810	10 050	665	10 890	620	11 400	590
2000.......	1.9	13 650	5460	605	7 980	555	9 060	535	9 480	525
4000.......	1.9	20 600	7320	925	10 590	865	11 670	840	12 120	835

[a] Minus sign indicates tension.

NOTE.—Flow tests series No. 8. Specimens, reinforced-concrete columns 5 in. in diameter by 48 in. long. Aggregate, 0 to ¾-in. local gravel. Fineness modulus, 4.47. Cement, normal portland. Aggregate-cement ratio, by weight (*a*) for 2000-lb. concrete, 8.40; (*b*) for 4000-lb. concrete, 4.80. Water-cement ratio, by weight (*a*) for 2000-lb. concrete, 0.83; (*b*) for 4000-lb. concrete, 0.53. Consistency, 5-in. slump. Curing, 21 days moist at 70 F. (21 C.), then stored as indicated. Age at loading, 28 days. Column loads determined in accordance with provisions of 1924 Report of Joint Committee on Specifications for Concrete and Reinforced Concrete.

steel has not greatly increased during the period of sustained load, under dry conditions for the columns with the smaller percentage of reinforcement the stress in the steel has increased more than five fold and for the columns with the larger percentage of reinforcement the concrete is actually in tension. This perhaps points to the desirability of modifying the formulas used in the design of columns to take into consideration the difference between wet and dry conditions.

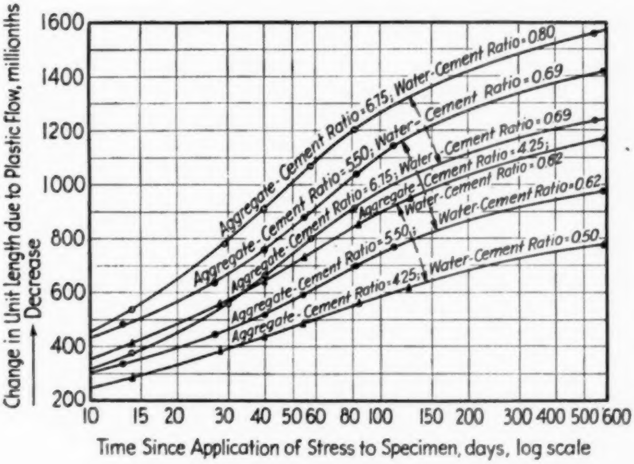

FIG. 2.—Effect of Aggregate-Cement Ratio and Water-Cement Ratio Upon Flow.

Flow tests, series No. 17. Specimens, 4 by 8-in. cylinders. Aggregate, 0 to $\frac{3}{4}$-in. local gravel. Fineness modulus = 4.94. Cement, normal portland; specific surface 1300 sq. cm. per g. Aggregate-cement ratio by weight, as indicated. Water-cement ratio by weight, as indicated. Consistency, variable. Curing, in fog at 70 F. (21 C.) for 28 days. Age at loading, 28 days. Sustained stress, 800 lb. per sq. in. compression. Storage after loading, in air at 70 F. (21 C.) and 50 per cent, relative humidity.

Effect of Aggregate-Cement Ratio and Water-Cement Ratio upon Plastic Flow:

Evaluation of the effects of variations in the aggregate-cement ratio and in the water-cement ratio upon plastic flow has frequently been complicated by the fact that these ratios have been varied simultaneously. In order directly to determine the effects of these factors, a series of tests was undertaken in which for concretes of each of three different cement contents, two different water-cement ratios were employed. All specimens were subjected to a sustained compressive stress of 800 lb. per sq. in. at the age of 28 days. The concrete was cured under standard moist conditions until time of loading, after which the tests were carried out in air at 50 per cent relative humidity and 70 F. The results of the tests are shown in Fig. 2.

It will be noted that the effect of water-cement ratio upon plastic flow is marked; for example, with a concrete of moderate cement content (aggregate-cement ratio = 5.50 by weight) subjected to sustained load for one year, a flow of about 930 millionths was obtained with a water-cement ratio (by weight) of 0.62, while with a water-cement ratio of 0.69, the flow was 1370 millionths, or about 50 per cent greater than that for the lower water-cement ratio.

For concretes having the same water-cement ratio, the richer mixes exhibited more flow than the leaner mixes. This is what might be expected if the major portion of the flow is considered to be due to movement in the hardened paste. It is noteworthy that for these tests, the percentage difference in paste content of two mixes having the same water-cement ratio was practically the same as the percentage difference in plastic flow of the same two mixes. For the concretes having a water-cement ratio of 0.62 by weight, the ratio of the absolute volume of the paste in the rich mix to that in the lean mix was about 1.19, and the ratio of the plastic flows for these two concretes was of the order of 1.22. For the concretes having a water-cement ratio of 0.69, the ratio of absolute volumes of pastes was about 1.16, and the ratio of the plastic flows was of the order of 1.12.

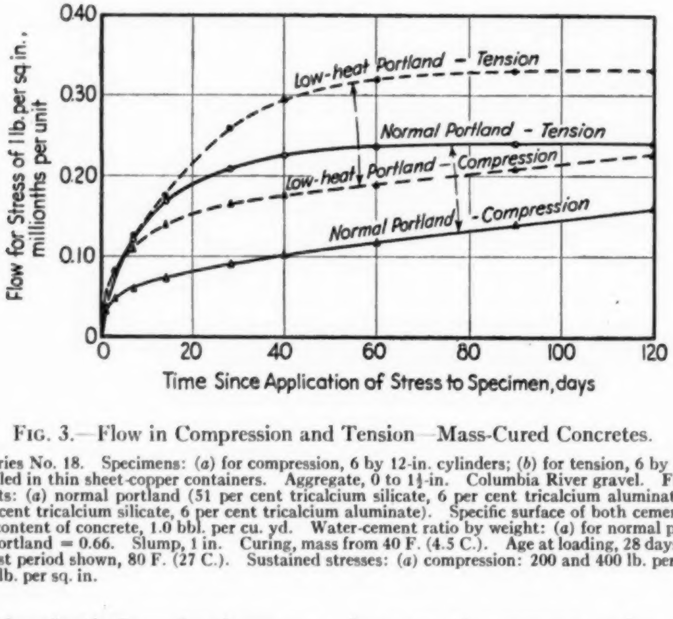

FIG. 3.—Flow in Compression and Tension—Mass-Cured Concretes.

Flow tests, series No. 18. Specimens: (*a*) for compression, 6 by 12-in. cylinders; (*b*) for tension, 6 by 33-in. cylinders. All specimens sealed in thin sheet-copper containers. Aggregate, 0 to 1½-in. Columbia River gravel. Fineness modulus = 5.46. Cements: (*a*) normal portland (51 per cent tricalcium silicate, 6 per cent tricalcium aluminate), (*b*) low-heat portland (26 per cent tricalcium silicate, 6 per cent tricalcium aluminate). Specific surface of both cements, 1800 sq. cm. per g. Cement content of concrete, 1.0 bbl. per cu. yd. Water-cement ratio by weight: (*a*) for normal portland = 0.69; (*b*) for low-heat portland = 0.66. Slump, 1 in. Curing, mass from 40 F. (4.5 C.). Age at loading, 28 days. Loading and storage during test period shown, 80 F. (27 C.). Sustained stresses: (*a*) compression: 200 and 400 lb. per sq. in.; (*b*) tension: 50 and 100 lb. per sq. in.

Statements have been made that (1) lean concretes flow *more* than rich concretes, and (2) weak concretes flow more than strong concretes. These statements require some qualification. For the same consistency, a lean (weak) concrete requires a higher water-cement ratio than a rich (strong) concrete; under these conditions the effect of increased water-cement ratio may more than offset the effect of the decrease in paste content, so far as plastic flow is concerned.

It seems reasonable to suppose that within ordinary limits the water-cement ratio has no marked effect upon the amount of the water of adsorption but that a higher water-cement ratio, through increasing the size of the pores between the cement grains, produces a paste structure from which the free water may the more readily escape, and hence, under conditions of stress the more readily may the water of adsorption be given up. Also, it appears reasonable to suppose that the more open the pore structure, the less will be the areas of contact between neighboring hydrated cement grains, and hence under a given load the higher will be the stresses over such areas of contact. There appears the interesting possibility that the particle-size distribution of a cement, through its effect upon the structure of the hardened cement paste, may have an important influence upon the rate and magnitude of volume changes in general.

Flow in Axial Tension and Compression:

The results of a series of tests to study the flow of concrete under sustained tension and compression are shown in Fig. 3. Two cements, a low-heat portland and a normal portland, are compared. Prior to the time of loading, the specimens were maintained under mass-curing conditions from a starting temperature of 40 F. The load was applied at the age of 28 days. During the period of test under sustained load, the temperature was 80 F. The specimens were stored continuously in sealed thin-copper containers to prevent loss of moisture.

The tension specimens were subjected to constant sustained stresses of 50 and 100 lb. per sq. in., and the compression specimens were subjected to constant sustained stresses of 200 and 400 lb. per sq. in. In order to make direct comparison between tensile and compressive flows, the changes in length have been reduced to "flow per lb. per sq. in." It is believed that the error involved in the assumption that flow is proportional to stress is not of sufficient magnitude to be of importance in these comparisons.

It is noteworthy that for these tests the rate of flow in tension for a period of two or three weeks after load application is markedly greater than the rate of flow in compression, resulting in a tensile flow per unit stress for the period shown (4 months), which was greater than the corresponding compressive flow. However, after a few weeks under load, the rate of tensile flow was considerably less than the rate of compressive flow. This points to the possibility that the compressive flow may in time equal or exceed that in tension. The stress-strain data from rapid-load tests of specimens made and stored under the same conditions as the flow specimens showed the same trend—a consistently larger tensile unit deformation than compressive unit deformation for the same unit stress.

The results of a later series of tests exhibiting similar trends are shown in Fig. 4. For this series, the concrete was made with a normal portland cement

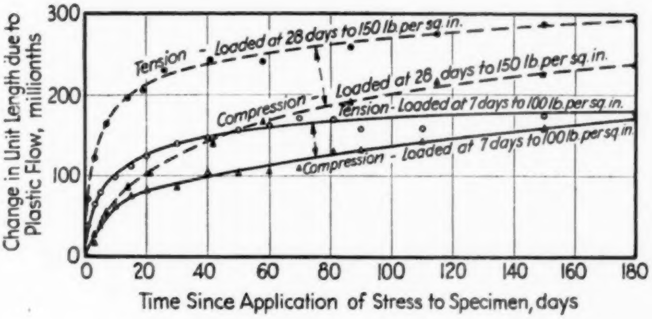

FIG. 4.—Flow in Compression and Tension—Standard-Cured Concrete.

Flow tests, series No. 20. Specimens: (*a*) for compression, 6 by 12-in. cylinders; (*b*) for tension, 6 by 33-in. cylinders Aggregate, 0 to ¾-in. local gravel. Fineness modulus = 4.57. Cement, normal portland. Aggregate-cement ratio by weight = 6.00. Water-cement ratio by weight = 0.63. Slump, 3 in. Curing, in fog at 70 F. (21 C.) until time of loading. Age at loading, as indicated. Sustained stress, as indicated. Storage after loading, in air at 70 F. (21 C.) and 50 per cent relative humidity.

and was moist cured until time of loading, after which the storage was in air of 50 per cent relative humidity at 70 F. For the specimens loaded at age 7 days, the sustained stress was 100 lb. per sq. in. in both tension and compression. For the specimens loaded at age 28 days, the sustained stress was 150 lb. per sq. in.

While this property of flowing more in tension than compression may not be characteristic of all concretes, it obviously is a desirable property, since it tends to reduce cracking either due to drying or to thermal changes. It perhaps partially explains the absence of cracks and checks in thin structures subjected to drying which were made with cements exhibiting above-normal unrestrained shrinkage. It perhaps explains why mass structures made with slow-hardening cements exhibit so few cracks as compared with corresponding structures in which more rapid-hardening cements have been employed.

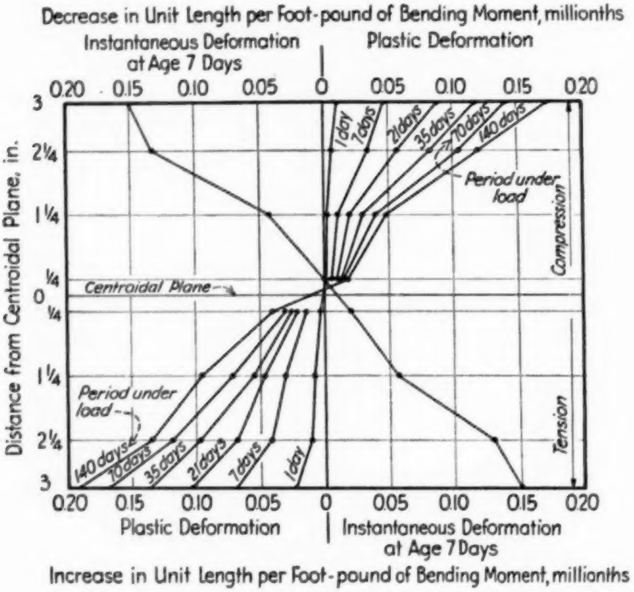

FIG. 5.—Fiber Strains Under Constant Sustained Bending Moment.

Flow tests, series No. 23. Specimens, 4 by 6 by 72-in. plain concrete beams. Aggregate, 0 to ¾-in. local gravel. Fineness modulus = 4.71. Cement, normal portland. Aggregate-cement ratio by weight = 6.00. Water-cement ratio by weight = 0.60. Slump, 2 in. Curing and storage during test, in fog at 70 F. (21 C.). Loaded beam supported at ⅓ points of a 69-in. length and loaded at ends. Loading schedule as follows:

Age, days	Total Load Applied at Each End, lb.	Bending Moment Produced, ft-lb.	Extreme Fiber Stress, lb. per sq. in.
7 to 28	100	192	96
28 to 30	250	480	240
30 to 148	350	670	336

Fiber Strains Under Sustained Flexural Load:

In Fig. 5 are shown fiber strains due to plastic flow alone at varying distances from the centroidal surface in a beam subjected to sustained bending moment under moist storage conditions. Load was applied at the age of 7 days and fiber strains were measured over a gage distance of 10 in. within a length over which the moment was uniform.

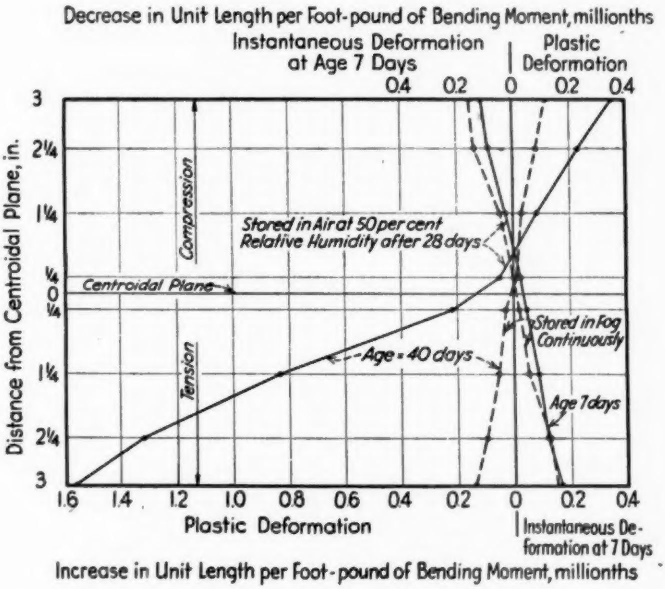

FIG. 6.—Comparison of Fiber Strains in Beams Under Constant Sustained Moment in Moist and Dry Storage.

Flow tests, series No. 23. Specimens, 4 by 6 by 72-in. plain concrete beams. Aggregate, 0 to ¾-in. local gravel. Fineness modulus = 4.71. Cement, normal portland. Aggregate-cement ratio by weight = 6.00. Water-cement ratio by weight = 0.60. Slump, 2 in. Loaded beams supported at ⅓ points of a 69-in. length and loaded at ends. Dry beams in air of 50 per cent relative humidity at 70 F. (21 C.) after 28 days in 100 per cent relative humidity at 70 F. (21 C.); loading as follows:

Age, days	Total Load Applied at Each End, lb.	Bending Moment Produced, ft-lb.	Extreme Fiber Stress, lb. per sq. in.
7 to 28	100	192	96
29 to 41	250	480	240

For storage and loading conditions of moist-stored beams, see Fig. 5.

It will be observed that the plastic flows of fibers on the tension side of the beam tend to be somewhat greater than corresponding flows on the compression side. These findings parallel those for flow in direct tension and compression. Also, it will be noted that the neutral surface remains substantially in a fixed position throughout the period of the test.

In a parallel series of tests, beams which were similarly loaded at the age of 7 days were removed from moist storage to dry air (50 per cent relative humidity) at the age of 28 days. Failure took place at the age of 41 days, when the calculated modulus of rupture was 240 lb. per sq. in. The modulus of rupture of a companion beam (stored under the same conditions but not previously subjected to bending) tested at the same age was 470 lb. per sq. in.—about 15 per cent less than that of a similar beam stored in moist (100 per cent relative humidity) air.

The plastic behavior of the beam stored in dry air after 28 days as compared with that of the beam continuously stored in moist air is shown by the diagrams of Fig. 6. It will be observed that at the age of 40 days, for the beam stored in dry air, the plastic flow of the extreme compression fiber was three times as great, and of the extreme tension fiber ten times as great as corresponding flows observed in the beam stored in moist air.

TABLE II.—FLOW OF MASS-CURED CONCRETES MADE WITH NORMAL AND LOW-HEAT PORTLAND CEMENTS.

Age at Loading, days	Cement	Flow per lb. per sq. in., millionths per unit length				
		Period Under Sustained Load, days				
		3	7	28	60	120
7	Normal	0.18	0.29	0.41	0.47	0.53
7	Low-heat	0.32	0.50	0.80	0.90	0.95
28	Normal	0.05	0.06	0.09	0.12	0.16
28	Low-heat	0.09	0.11	0.16	0.19	0.22

NOTE.—Flow tests, Series No 18. Specimens, 6 by 12 in. cylinders sealed in thin-copper containers. Aggregate, 0 to 1½-in. Columbia River gravel. Fineness modulus, 5.46. Cements: (*a*) normal portland (51 per cent tricalcium silicate, 6 per cent tricalcium aluminate); (*b*) low-heat portland (26 per cent tricalcium silicate, 6 per cent tricalcium aluminate). Specific surface of cements, 1800 sq. cm. per g. Cement content of concrete, 1.0 bbl. per cu. yd. Water-cement ratio by weight: (*a*) normal portland, 0.69; (*b*) low-heat portland 0.66. Slump, 1 in. Curing, mass from 40 to 80 F., and then constant at 80 F. Normal portland reached 80 F. at 10 days, low-heat portland reached 80 F. at 20.5 days. Loaded to sustained compressive stresses: (*a*) for normal portland: 100 and 200 lb. per sq. in. at 7 days; 200 and 400 lb. per sq. in. at 28 days; (*b*) for low-heat portland: 100 lb. per sq. in. at 7 days; 100, 200, and 400 lb. per sq. in. at 28 days.

The results of the two groups of tests just described demonstrate the very marked effect of the humidity of the surrounding air upon both the plastic flow and upon the modulus of rupture of concrete subjected to sustained bending moment. Obviously, whether the structural element be plain or reinforced, it is desirable that the concrete of which it is composed be of a quality which under drying conditions will exhibit maximum flexural strength under sustained moment. In other words, it is desirable that the concrete be one in which the effects of plastic flow and shrinkage due to drying, one tending to offset the other, would combine to produce the most favorable distribution of stresses.

Effect of Fineness and Composition of Cement upon Plastic Flow:

The results of observations of the plastic flow of mass-cured concretes made with a normal portland and a low-heat portland cement, each ground to a specific surface of 1800 sq. cm. per gram, are shown in Table II.

The temperature conditions under which each concrete was stored were different, so that the rate of hardening was dependent upon the curing conditions as well as the type of cement. The values of flow at each of the several stresses under which the tests were made have been reduced to flow per pound per square inch and averaged to obtain the values shown. Under these conditions, the low-heat portland generally exhibited a greater flow than the normal portland cement, on the average by about 80 per cent. The magnitude of flow of the concretes loaded at 7 days was greatly in excess of that of the concretes loaded at 28 days.

The results of a series of tests undertaken to study the effect of the composition and fineness of the cement upon flow in axial compression are shown in Fig. 7. There were employed portland cements of two chemical compositions, one of normal and one of the low-heat type, each ground to specific surfaces of 1300 and 2200 sq. cm. per gram. The specimens were loaded in axial compression to 800 lb. per sq. in. after 28 days of standard moist curing, and were stored during the period of test in air of 50 per cent relative humidity at 70 F.

It will be observed that the coarsely-ground low-heat cement exhibited much the greater plastic flow at all ages and that its rate of flow was greatest not only at the early ages but also at the late ages.

Certain European experience has pointed to the desirability of employing relatively coarse low-heat cements for mass structures, in order to reduce cracking to a minimum. The Crystal Springs Dam, south of San Francisco, built nearly fifty years ago with a coarse cement of composition quite similar to that of our modern low-heat cements, is perhaps one of the finest examples of our older structures, located in a comparatively dry climate, that has developed almost no cracks nor surface checks. The same general freedom from cracks is also observed in the recently constructed Morris Dam, where the modern type of low-heat cement was employed for the first time in this country. On the other hand, where modern, more rapid-hardening portland cements of normal or modified composition and comparatively high fineness have been employed, mass structures have shown frequent cracks of considerable magnitude. It is believed that this difference in degree of cracking in service structures under field conditions can be accounted for in no other way than through differences in the plastic flow characteristics of the concretes of which these structures are composed, primarily due to differences in composition of cement.

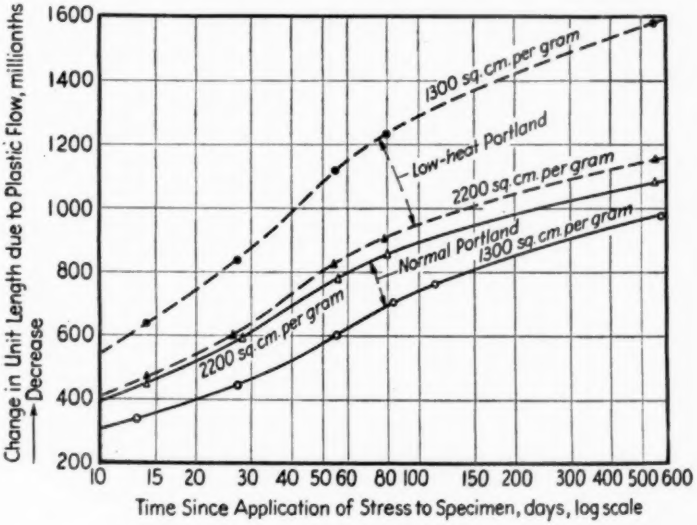

FIG. 7.—Effect of Composition and Fineness of Cement upon Flow.

Flow tests, series No. 17. Specimens, 4 by 8-in. cylinders. Aggregate, 0 to $\frac{3}{4}$-in. local gravel. Fineness modulus = 4.94. Cement: (*a*) normal portland (52 per cent tricalcium silicate, 25 per cent dicalcium silicate, 8 per cent tricalcium aluminate, 13 per cent calcium ferro-aluminate); specific surface, as indicated; and (*b*) low-heat portland (30 per cent tricalcium silicate, 47 per cent dicalcium silicate, 8 per cent tricalcium aluminate, 12 per cent calcium ferro-aluminate); specific surface, as indicated. Aggregate-cement ratio = 5.50 by weight. Water-cement ratio = 0.62 by weight. Consistency, variable. Curing, in fog at 70 F. (21 C.) for 28 days. Age at loading, 28 days. Sustained stress, 800 lb. per sq. in. compression. Storage after loading, in air at 70 F. (21 C.), and 50 per cent relative humidity.

Again referring to Fig. 7, it is seen that in the case of the low-heat portland, the flow is greater for the coarser cement; in the case of the normal portland cement, the reverse is true. The reason for the reversal is not evident, but it seems possible that it may be due to variations in the particle-size distribution of the cements, which variations are not manifest in the calculated values of the specific surface.

The results of a later series of tests covering a wider range in specific surface of cements exhibit similar peculiarities, again pointing to the possibility that the particle-size distribution of a cement may exert a marked influence upon the flow characteristics of concrete.

Thermal Stress Studies:

To investigate the thermal stresses developed in concrete under temperature conditions similar to those occurring in mass concrete, tests were made on a pair of 12 by 48-in. concrete cylinders which were maintained at constant length through the period of test. The concrete contained 1.0 bbl. of cement per cubic yard, aggregate of 3-in. maximum size, and had a slump of 3 in. The placing temperature was 57 F., and the temperature of the testing room was maintained in accordance with a predetermined time-temperature schedule. The temperatures throughout the period of test, as measured at the center of the specimens, are shown by the lower diagram of Fig. 8. The specimens were sealed in thin-copper containers. Provision was made, and the testing apparatus was designed, so that either compressive or tensile stresses could be sustained. Observations, by means of which the length of each cylinder was controlled, were made through the use of a strainmeter located at the center of the specimen along its longitudinal axis, and check measurements were made through the use of a 20-in. strain gage applied to three gage lines on the surface of the specimen. To determine the changes in length due to thermal changes alone, 1-ft. concrete cubes in sealed metal containers were cast at the same time as the cylinders. The length changes of the cubes were measured by both internal strainmeter and strain gage applied to surface gage lines. Throughout the period of test, the

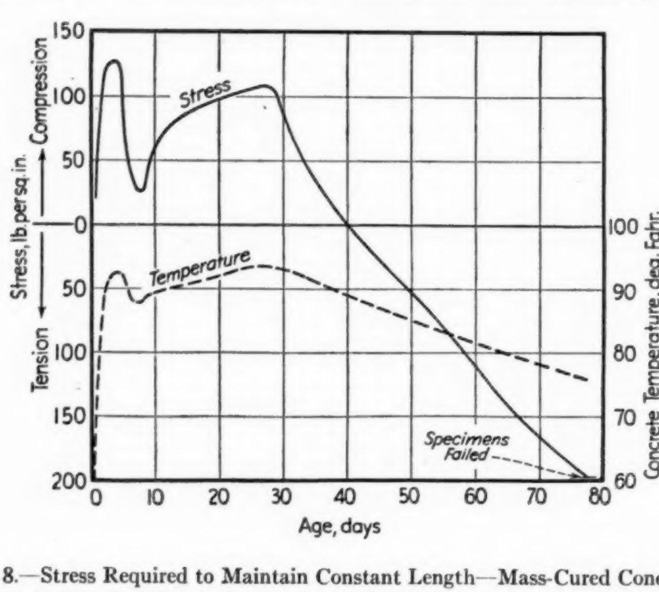

FIG. 8.—Stress Required to Maintain Constant Length—Mass-Cured Concrete.

Flow tests, series No. 19. Specimens, 12 by 48-in. cylinders sealed in thin copper containers. Aggregate, 0 to 3-in Columbia River gravel. Fineness modulus = 6.10. Cement, portland puzzolan. Cement content of concrete = 1.0 bbl per cu. yd. Water-cement ratio by weight = 0.63. Slump, 3 in. Curing, mass from 57 F. (14 C.)—temperature as indicated by curve. Age at loading, 14 hr. Stress, as indicated by curve—specimen maintained at length equal to that observed at age 6 hr. Stress and temperature determined from observations on Carlson electric strainmeters.

temperatures of the unrestrained cubes were the same as those of the test cylinders.

Based on information obtained from auxiliary tests to determine strength and plastic flow of the concrete at various intervals after casting, load was slowly applied to the large cylinders at the age of 14 hr. After sufficient load had been applied to bring the specimen to the length that had been observed at age 6 hr., the specimens were maintained at this length by periodically adjusting the load, which was measured by means of calibrated coil springs.

The stresses required to maintain constant length, averaged for the two cylinders, are shown in Fig. 8. During the first 4 days, as the temperature rose 35 deg. Fahr., the stress increased to about 130 lb. per sq. in., compression. This stress divided by the thermal expansion which took place in the unrestrained concrete cubes during the period, gives a sustained modulus of elasticity of about 900,000 lb. per sq. in. A weighted average value of the modulus of elasticity, determined from rapid-load tests upon auxiliary specimens during this period, lies between 1,500,000 and 2,000,000 lb. per sq. in. Thus, it is evident that, due to plastic flow, the stresses were reduced by perhaps 50 per cent of what they would have been if flow had not taken place.

During the period 4 to 7 days, when the temperature dropped about 4 F., the stress decreased about 100 lb. per sq. in. If this reduction in stress is divided by the thermal contraction which took place in the unrestrained cubes during the period of temperature drop, there is obtained a value of approximately 5,000,000 lb. per sq. in., which may be considered as the effective modulus of elasticity during the period of decreasing temperature. The corresponding modulus of elasticity of concretes, as determined from rapid-load tests on auxiliary cylinders, was of the order of 2,500,000 lb. per sq. in. It is noteworthy that as stress is released after a period of sustained load, the effective modulus of elasticity is much greater than would be obtained by the ordinary procedure of determining deformations under rapid-loading conditions where permanent set is included in the measured deformations.

During the period 7 to 26 days, when the temperature rise was 6 deg. Fahr., the stress increase was 80 lb. per sq. in. The ratio of this stress to the thermal expansion of the unrestrained cubes during the same period was about 2,000,000 lb. per sq. in.; during this period the average value of the modulus of elasticity determined from rapid-load tests on auxiliary specimens was of the order of 3,000,000 lb. per sq. in.

When the temperature had declined from the maximum of 94 F. to 89 F. (a drop of only 5 deg. Fahr.), the compressive stress in the restrained specimen had declined to zero. When the temperature had declined to 76 F. (19 deg. Fahr. above the initial temperature), the specimen failed in tension at a stress of approximately 200 lb. per sq. in.

Over the period, age 26 days to age 77 days, during which the stress declined 310 lb. per sq. in., the effective modulus of elasticity was 3,800,000 lb. per sq. in. as compared with a modulus of 3,600,000 lb. per sq. in. determined from rapid-load tests at the age of 28 days. The relatively high value of the effective modulus is due in part to the conditions existing during the release of stress, as noted previously.

While no concrete structure is completely restrained and therefore the thermal stresses developed during the course of the experiment just described were in excess of those which would actually be set up in a structure built of the given concrete and subjected to the given temperature changes, yet in the cases of thick walls, heavy foundations, bridge piers, and dams resting on rock foundations, the conditions of the experiment may be approached. For this reason the results are of value as indicating in many mass structures the existence of high stresses due to heating and cooling, not only in the vicinity of the foundation rock but at exposed faces, keyways, and along construction-joint planes between lifts. In certain types of structures these residual stresses due to thermal changes may not be a matter of importance. In the case of dams, however, these stresses, in combination with those due to load, may produce a stress system far different from that calculated by the usual methods of analysis, which entirely ignore the effects of thermal changes. The condition of dams, where there are in evidence many structural cracks in spite of contraction joints, makes it appear that this must be true.

The results are also of value in showing the large part that plastic flow plays in the development of stress during the heating-cooling cycle and in indicating a method which may be employed in experimentally determining the magnitudes of thermal stresses that are likely to occur in any proposed structure. When through the results of extensive experiment there have been established the time-flow-stress-temperature-restraint relationships for a wide variety of conditions, they may be employed in a rational method of thermal-stress analysis. This appears to be the next step—a step which will remove an important element of uncertainty which now exists.

Acknowledgment:

These investigations have been made possible through funds contributed by the University of California and the Engineering Foundation, and certain of the investigations were made in cooperation with the Corps of Engineers, U. S. A.

Among those who have rendered valuable services in carrying out the investigations are the following persons: G. E. Troxell, Associate Professor of Civil Engineering, J. L. Banville, Assistant in the Engineering Materials Laboratory, and T. A. Amneus, J. Brummer, E. Daigle, H. Degenkolb, J. W. Gerhart, W. Giddings, A. F. Liebscher, and O. F. Weymouth, former engineering students, all of the University of California.

DISCUSSION

Mr. D. E. Parsons.[1]—What is the effect of the moisture content of the concrete at the time of loading on the plastic flow of concrete stored in air subsequent to loading?

Mr. R. E. Davis.[2]—It depends greatly upon the amount of moisture present in the concrete at the time of loading. In concretes which are thoroughly dry, the rate of flow is comparatively small, but in the usual case of dry air storage where the concrete at the time of loading is moist and subsequently dries out, the rate of flow is large. I suppose this is due to the fact that the surface of a column, for example, shrinks much more rapidly than does the interior. Under load there is produced a comparatively high compressive stress near the center; while at the same time the surface fibers may actually be in tension. In building columns under load I have observed minute horizontal cracks, indicating the existence of such tensile stresses.

Mr. L. W. Teller.[3]—I should like to ask Mr. Davis whether he has observed in any of the specimens a progressive increase in volume with time, that is, a growth in the volume of the concrete not due to stress conditions. I have in mind particularly wet storage conditions.

Mr. Davis.—We have observed that with any concrete there is an increase in volume with time under moist storage conditions. Of course, the flow in the case of a column may more than offset, and usually does more than offset, the increase in volume due to water adsorption.

Mr. Teller.—It occurred to me that this might be a complicating factor in some of the observations that you are making. If there were volume changes of that sort occurring, an error of unknown magnitude is introduced which would affect both the apparent tensile deformations and the apparent compressive deformations.

Mr. Davis.—What you say is true unless the concrete before loading has reached a state of volume equilibrium. However, in the investigations here reported the length changes of unloaded companion specimens have been observed, and the average plastic flow has been determined by taking the difference between the length change of a loaded specimen and the length change of a corresponding unloaded specimen.

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THE WICK TEST FOR EFFLORESCENCE OF BUILDING BRICK[1]

By J. W. McBurney[2] and D. E. Parsons[2]

Synopsis

This paper describes the wick test for efflorescence on building bricks and presents data showing the relation between results of wick tests and the appearance of efflorescence on exposed masonry panels. It is concluded that the results of the 5-day wick test correlate well with the grading on exposed panels when the efflorescence on individual bricks rated in excess of "moderate" on the scale used for grading intensity of visible efflorescence.

If a brick is set on end in shallow water and that portion of the brick above the water is exposed to conditions favoring evaporation, water entering the brick by capillarity will move upward and outward through the brick. The water will tend to dissolve soluble salts in the brick and transport them to the surface where they may be progressively deposited as the water, in which they are dissolved, evaporates. These phenomena have been variously applied in a "wick test" which may be regarded as a test to indicate the presence of soluble salts which may contribute to efflorescence on masonry.

The authors of this report do not know who first used this test for efflorescence but one of the authors tested brick by this method in 1922. Palmer[3] reported: "The brick were set vertically in the pan and in about 1 in. of distilled water, and the tests in all cases were continued for a period of six months." Butterworth[4] used a wick test with exposure from two to three weeks. In 1930, 5-day wick tests were made on 684 bricks, each of a different grade or brand, from 255 manufacturers. Although the results of these tests were not published they were given to the manufacturers supplying the samples. Since 1930 considerable use of the wick test has been made by the National Bureau of Standards at the request of certain Government construction agencies.

In the late summer of 1936 a number of small panels or wallettes were constructed from 18 types of brick for the purpose of studying the effects of weather on brick masonry, including efflorescence. Samples of the brick used in constructing these panels were subjected to the 5-day wick test. The relation between the results of this wick test on the bricks and the amount of apparent efflorescence on these panels is considered in this paper.

The Wick Test for Efflorescence

At the National Bureau of Standards, the wick test consists in standing a whole

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[1] Publication approved by the Director of the National Bureau of Standards of the U. S. Department of Commerce.

[2] Senior Technologist, and Chief of Masonry Construction Section, respectively, National Bureau of Standards, Washington, D. C.

[3] L. A. Palmer, "Cause and Prevention of Klin and Dry-House Scum and of Efflorescence of Face-Brick Walls," *Technologic Paper No. 370*, Nat. Bureau Standards, (1928).

[4] B. Butterworth, "Contributions to the Study of Florescences, VI," *Transactions*, Ceramic Soc. (London), Vol. 32, p. 270 (1933).

brick on end in 0.5 in. of distilled water, maintained at approximately a constant level by inverting a flask of water over the tray, with the mouth of the flask held at the desired level. The brick, except that portion in contact with water, is exposed to the air of the laboratory at ordinary temperature. At the end of 5 days the brick is dried in an oven at 105 to 110 C. for at least 18 hr. and the amount of efflorescence is estimated by visual comparison with an untreated specimen.

The amount of efflorescence is rated on a scale consisting of 6 classifications or grades, described as follows:

(0) *None*—No observable difference in the appearance of a brick after test and before.

(1) *Trace*—Efflorescence barely distinguishable by careful comparison.

(2) *Slight*—Observable. Not sufficient efflorescence to affect materially the appearance when viewed at a distance of approximately 6 ft.

(3) *Moderate*—Distinct coating but the original color of the brick distinguishable under the efflorescence.

(4) *Considerable*—The original color of the brick masked by the efflorescence.

(5) *Abundant*—Efflorescence in such quantity that it may be brushed off readily.

It is considered that Butterworth's[4] first grade, "Bricks that are not liable to eflorescence, that is, those on which the efflorescence developed in the test is nil or negligible" corresponds to the "none," "trace" and "slight" of this grading. His second grade "Bricks that can only be recommended with reserve from the point of view of efflorescence" represents "moderate" and possibly "considerable" and the third grade, "Bricks the use of which would constitute a disaster" corresponds to "abundant."

In the wick test on a particular brick, the amount of efflorescence that appears on the surface is affected by several factors other than the quantity of salts per unit of volume of the brick.[5] For example, if the brick has a very low rate of absorption, approximate equilibrium will be quickly reached between water evaporated and water diffusing through the brick by capillarity. Efflorescence will, under these conditions, appear at the junction between the damp and dry portions of the brick, usually as a horizontal band around the brick. On one specimen, this band was less than one inch above the surface of the water. Obviously the dry portion of this brick did not contribute to the efflorescence. Again, there are certain combinations of temperature, humidity, circulation of air, and pore structure in which evaporation will take place beneath the surface of the brick with the result that some salts will be deposited there and hence will not be visible. This is the "cryptoflorescence" referred to by Cooling.[6] Then, too, if the brick specimen has a large total absorption, a high rate of absorption and contains a moderate amount of readily soluble salts, the efflorescence obtained in the wick test may appear as beads on the four upper corners of the brick instead of being distributed over the surface.

EXPOSURE TESTS OF PANELS OF BRICK MASONRY

The panels considered in this study of the relations between the results of the wick test and the actual exposures were made of 18 brands of brick (briefly described in Table I) and a single kind of mortar (1.0 part portland cement, 0.1 part hydrated lime, 3.0 parts sand, by weight). All panels are 4 ft. high, 3 ft.

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[5] If standardization of the wick test is to be attempted, one might begin by prescribing the temperature, humidity and velocity of air and then working either for a definite time or until a definite quantity of water has been evaporated.

[6] L. F. Cooling, "Contributions to the Study of Florescence. II Evaporation of Water from Brick," *Transactions*, Ceramic Soc. (London), Vol. 29, No. 2, pp. 39–52 (1930).

long and 12 in. thick and are set vertically 12 in. deep in the ground. Common bond was used throughout and the workmanship was characterized by furrowing of the horizontal joints and a minimum filling of the vertical joints.

Each brand of brick (except No. 14) was used to build four panels, each of which was "protected" differently, as follows:

1. No protection.
2. Top of panel covered with loosely fitting sheet-copper cap.
3. Sheet-copper "flashing" in horizontal joint through the panel, 2 in. above ground level.
4. Combination of 2 and 3 (see Fig. 1).

The panels are placed on the top of a gradually sloping and somewhat wooded knoll in the northwest corner of the Bureau's grounds at Washington, D. C. This site was selected so that all panels would be exposed to essentially the same atmospheric conditions. The individual panels, facing east and west, were spaced 5 ft. apart between faces and 4 in. apart between ends.

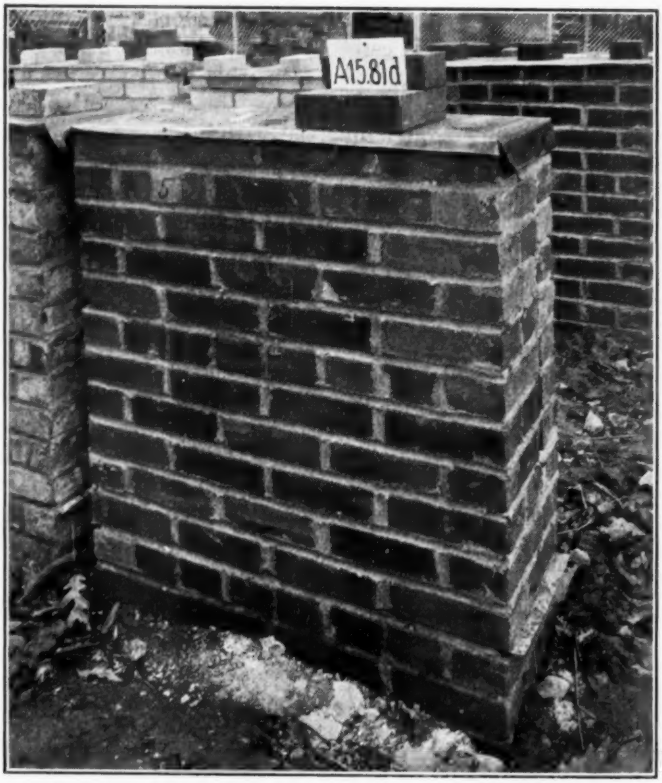

Fig. 1.—Panel of Brick 2 Showing Sheet Copper Cap over Top and Sheet Copper Flashing Above Ground (Protection 4).

Photograph taken November 25, 1936.

Random samples consisting of 12 specimens each of the 18 brands of brick used in the construction of these panels were subjected to the 5-day wick test.

RELATION BETWEEN INDICATIONS OF WICK TESTS ON BRICK AND EFFLORESCENCE ON EXPOSED MASONRY PANELS

The masonry panels were inspected on November 30, 1936, and again on March 4, 1937. The first inspection was made six weeks after completion of construction of the panels. There had been no rain for two weeks preceding this inspection. Subsequent rains temporarily removed all traces of visible salts from the panels. The second inspection took place on the fourth day of clear weather.

The selection of the ratings for efflorescence on the panels was not as simple as for individual bricks because the ratings assigned to the panels depended to a far greater extent upon the judgment of the inspector. Both intensity and distribution of the efflorescence were considered. As indicated by footnotes in Table I, some of the bricks in certain panels of bricks 5 and 16 showed much more efflorescence than others during the first inspection. At the time of the second inspection, the efflorescence was more uniformly distributed. In attempting to evaluate or visualize the appearance of the panels, it might be stated that ratings less than "moderate" (3) indicate no material change in the appearance of masonry when viewed from a distance of 20 ft.

TABLE I.—COMPARISON OF RESULTS OF 5-DAY WICK TESTS FOR EFFLORESCENCE OF BRICK AND EFFLORESCENCE RATINGS FOR MASONRY PANELS.

Brick	Wick Test Ratings[a]	Ratings for Masonry Panels[a]								Absorption Data[b]		Description of bricks[c]
		First Inspection				Second Inspection						
		Protection[f]				Protection[f]						
		1	2	3	4	1	2	3	4	C/B	B, per cent	
No. 1.....	0	4	3	3	3	2	2	2	2	0.69	24	SM; C; Salmon, Pennsylvania
No. 2.....	0	1	2	2	0	1	2	1	1	0.81	15	DP; C; Virginia
No. 3.....	0	1	1	0	2	1	1	1	1	0.71	18	Sand-lime, Pennsylvania
No. 4.....	0	2	1	3	2	1	1	1	1	0.71	12	SC; C; Maryland
No. 5.....	1	4	3	3	1[d]	2	2	2	2	0.74	17	SM; C; Hard, Pennsylvania
No. 6.....	1	2	1	2	1	3	3	3	3	0.75	15	EC; C; Chicago
No. 7.....	2	3	4	3	3	4	3	3	3	0.87	24	SM; C; Detroit
No. 8.....	2	3	4	3	4	3	2	1	3	0.91	33	SC; C; Wisconsin
No. 9.....	3	0	1	1	1	2	1	2	2	0.86	14	SC; S; Light hard, West Virginia
No. 10.....	4	4	3	3	1	4	3	4	3	0.88	14	DP; S; Texas
No. 11.....	4	4	4	4	3	4	4	4	3	0.83	21	SM; C; Hard, Hudson Valley
No. 12.....	4	5	4	4	4	3	4	3	3	0.90	23	SM; C; Salmon, Hudson Valley
No. 13.....	4	5	4	5	4	3	3	3	3	0.84	18	SM; C; Salmon, New England
No. 14.....	4	4	.....	.....	.....	4	.....	.....	.....	0.86	11	SC; S; Deaired, Pennsylvania
No. 15.....	0–2	0	1	2	0	2	2	2	1	0.57	5	SC; S; Hard, West Virginia
No. 16.....	0–5	2[e]	1[e]	3	0	3	2	3	2	0.64	16	SC; S; Minnesota
No. 17.....	1–3	3	2	2	2	2	1	2	1	0.78	33	DP; C; Texas
No. 18.....	2–3	1	1	2	2	2	2	2	1	0.84	12	SC; C; Salmon, Maryland

[a] For convenience, the rating are expressed numerically: 0 = none. 1 = trace. 2 = slight. 3 = moderate. 4 = considerable. 5 = abundant.

[b] C = water absorption in the 24-hr. submersion test and B = water absorption in the 5-hr. boiling test, described in the Tentative Methods of Testing Brick (C 67 - 36 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 780 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 432.

[c] SM = soft mud SC = side cut. EC = end cut. DP = dry press. C = clay. S = shale.

[d] Two bricks rated 3.

[e] One brick rated 4.

[f] Protection 1 = no protection; 2 = top of panel covered with loosely fitting sheet-copper cap; 3 = sheet-copper "flashing" in horizontal joint through the panel, 2 in. above ground level; 4 = combination of 2 and 3.

Table I includes the results of the wick tests, the results of these two inspections, and data on the absorption of the bricks. The reader is cautioned against ascribing any cardinal significance to the numerals used in rating efflorescence. It is probable that the difference in the amounts of salts warranting a change of rating from "moderate" (3) to "considerable" (4) was greater than the difference to change from "none" (0) to "moderate" (3).

The data pertaining to bricks 1 to 14 inclusive are grouped according to the ratings for these bricks in the wick test. The individual bricks (12 each) of each of these types were rated alike in that test. Those for bricks 15 to 18 inclusive showed significantly different amounts of efflorescence in the wick test, and the data for these are in a separate group.

The data of Table I, pertaining to bricks 1 to 14 inclusive, show the following:

1. When the rating of the bricks in the wick test was "considerable" (4), 16 of the 17 panels built from these bricks rated "moderate" (3) or more in the first inspection and all these rated "moderate" (3) or more in the second inspection.

2. When the rating of the bricks was "slight" (2) or less, 27 of the 32 panels of the bricks rated "moderate" (3) or less in the first inspection and 31 of the 32 rated "moderate" (3) or less in the second inspection.

3. When the rating of the bricks was "trace" (1) or less, 22 of the 24 panels rated "moderate" (3) or less in the first inspection, and in the second inspection all rated "moderate" (3) or less and 20 of the 24 rated "slight" (2) or less.

4. The data presented do not show that the kind of protection had a significant effect on the amount of efflorescence on the panels.

It is evident that the correlation between the indications of the wick tests on the bricks and the appearance of efflorescence on the masonry was somewhat better for the data of the second than for the first inspection. Possibly data obtained during future inspections will show a continuance of this trend and also a significant difference in the appearance of panels with different protections (2, 3, 4).

The panels built with bricks rating "none" (0) to "slight" (2) tended to rate one or two grades higher than the individual bricks. Some of the efflorescence on these panels probably was caused by soluble salts originally in the mortar. The appearance of the panels during the first inspection indicated that salts from the mortar had contributed to the deposit. The efflorescence on most of the panels appeared on the exposed faces of the bricks and was chalk white in color, whereas, that on some of the panels of bricks rating "none" (0) was limited largely to the joints and edges of the bricks adjacent thereto and was gray in color.

CONCLUSIONS

The results of the tests and comparisons showed that:

1. If considerable efflorescence appeared on bricks in the 5-day wick test, masonry panels built of bricks of the same brand showed an objectionable (moderate or more) amount of efflorescence.

2. If the efflorescence on bricks in the wick test was only a trace or less, most of the masonry panels built from the same brand of bricks showed only a slight (or less) amount of efflorescence.

3. Soluble salts from mortar appeared to have caused efflorescence on some of the panels.

DISCUSSION

MR. L. A. PALMER[1] (*presented in written form*).—In a considerable number of buildings, efflorescence is most noticeable on parapet walls beneath copings, above the lintels and below the sills of windows and below projecting brick courses which collect the wash of the wall. The evaporation of water which has entered through poorly constructed areas of a wall usually takes place at some joint below that at which it entered, the movement of water saturating the masonry materials being influenced both by capillary forces and by gravity. However, it also often happens that the movement of water containing the dissolved salts of efflorescence is in the upward direction from the ground level. Particularly is this true where quite porous bricks are used below grade. By interposing slate or other relatively impermeable material in the bed joint near the ground level, this upward movement of moisture may be effectively checked.

The observation of Cooling, of the staff of the British Building Research Station, is interesting. The development of "cryptoflorescence" in clay and shale building units is similar to the deposition of salts within the unit taking place during the early drying and firing periods of the manufacturing process. It would seem that in certain instances liquid water may change to water vapor within the body of the clay mass. When this happens, the salts do not reach the surface but are deposited in the interior. Ordinarily, one would consider that the conversion of liquid to vapor occurs only on the solid surface but apparently this is not always the case. When a "green" brick going into the dryer is pressed with the palm or fingers of the hand, one may frequently note, after drying, a scum deposit over the finger indentations, the remainder of the surface being relatively free from scum. The pressure of the hand reduces the number and size of capillary "exits." Consequently evaporation through such areas occurs relatively slowly and water must move entirely outward to the surface before there is appreciable evaporation. It may be possible that the capillary structure of a burned clay or shale building unit may sometimes be such as to permit the change of water liquid to water vapor within the solid as well as at its surface.

The soluble matter in the dry mortar ingredients is quickly dissolved when water is added. This soluble matter passes quickly into dry porous bricks by means of "suction." If the bricks are fairly impervious or if they are porous bricks set wet, the soluble matter tends to remain within the mortar and, upon evaporation, to be deposited in part on the exterior surfaces of the joints. In this connection it should be noted that most if not all mortar mixes produce solutions saturated with calcium hydroxide (lime) when the mixing water is added. Lime itself is soluble and as such may contribute noticeably to efflorescence. As efflorescence on the face of a wall, it is slowly changed to the carbonate and thereafter is removable only with acid solution.

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[1] Associate Chemist, U. S. Bureau of Public Roads, Washington, D. C.

The sulfates of soda, potash, lime, and magnesia comprise most of the white or gray efflorescence. A relatively rare type of efflorescence traceable to clay or shale building units is that produced by soluble vanadium compounds. This first appears as a brilliant yellow encrustation and gradually changes to green or blue and appears usually on certain light or buff-colored bricks. Chromate and molybdate salts, which are deposited even less frequently than the vanadates, are similar in color to the latter.

The writer knows of no test more practical or expedient than the wick test. It is possible to test a considerable and representative number of bricks by this procedure. The period of the test, 5 days, should be ample for all practical purposes.

Presumably, furrowing the horizontal joints and only partially filling the vertical ones in the test panels was a procedure followed in order to accelerate entrance of moisture and thus make the tests more positive, although the authors have not stated that this was the purpose of this procedure. It is a procedure that has been generally followed in actual construction and is one that cannot be denounced too vigorously. It should be economically possible to have reasonably well filled joints.

MR. C. C. CONNOR[2] (*presented in written form*).—It is very necessary that there be some method of determining, approximately at least, the amount of efflorescent salts in brick. Not only does efflorescence disfigure a building, but there appears to be some evidence that efflorescent salts may produce a result much more harmful, namely, that the action of these salts may cause brick to spall.

In a group of some fifty buildings which have been under my observation, six are showing a considerable amount of spalling of the face brick. The conditions at five of these buildings point towards the action of efflorescent salts as a possible cause of the spalling. These five cases divide themselves into two groups by the nature of the brick and the spalling action. One group includes two buildings having very similar fire-clay face brick with an impervious face formed in the burning without the addition of any glazing material. The lighter colors seem to be underburned; the darker colors appear to have been moderately well burned. Samples from the original shipments were tested chemically by a method that gave rough quantitative comparisons and were estimated to contain slight to moderate amounts of soluble sulfates. The efflorescence on the buildings has shown in considerable amounts in concentrated areas of limited extent, but over the greater area of the building it has not been generally noticeable.

Three years after the construction of these buildings, it was noted that the impervious face of the brick was beginning to disappear in certain areas, and this condition has continued to increase. The buildings are now 6 yr. old. The destruction of the brick face was not the result of large flakes being lifted off. The first action took the appearance of scratches on the face, followed by the development of small pits, and in the most advanced stage evident today, the impervious face has completely disappeared, leaving a rough surface with small pits. In no case has the disintegration progressed to any great depth, but the appearance has become decidedly unsatisfactory.

When first noticed, the spalling was confined to those areas where efflorescence appeared in considerable amounts. Even in individual bricks the

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[2] Assistant Engineer, New Jersey Bell Telephone Co., Newark, N. J.

areas where the face was destroyed coincided with concentrations of efflorescence. As the spalling spread, bricks were affected in areas where the efflorescence was slight, but numerous inspections during periods favorable to the formation of efflorescence showed that efflorescence had concentrated at the points where the spalling had occurred.

One of the interesting features of the spalling was that all of the noticeable increase and spread of the condition occurred in the spring months when there were no freezing temperatures. This coincided with the appearance of much efflorescence, but the amount was no more than appeared in the fall months. It would appear, however, that the spring season would be more favorable to cycles where the efflorescent salts could lose and take on molecules of water of crystallization between the periods of formation and dissolution by rain.

If efflorescence is a cause of spalling of brick, it is felt that the taking on of water of crystallization by an effloresced salt is more likely to be the cause of disintegration than the crystallizing of the salt out of solution. The ease with which the sulfates of sodium and magnesium lose and take on water of crystallization puts them under suspicion since, according to Palmer,[3] they are typical salts of brick efflorescence.

Samples of affected brick, including various stages of the destruction of the face, were cut out of the walls of one of the buildings in this group and subjected to a freezing-and-thawing test, but a long series of cycles failed to produce any definite increase in the spalling. Frost action evidently was not a cause in this case.

The second group consists of three buildings 7 to 8 yr. old, built with a common clay face brick of like manufacture but differing in size and surface texture. All of the bricks have a highly absorbent face and appear to be moderately well burned. None of them was tested for efflorescent salts before use. The efflorescence on the buildings has shown in abundant amounts on concentrated areas and has been present in variable amounts on other areas. At two of the buildings it has been widespread, but at the third it has been limited to the parapet walls.

The spalling at these buildings was not noticed until three years ago when the observed spalling at one building caused a survey to be made of others that had been recently constructed. The condition when first noticed was quite advanced in many areas and has continued to progress and spread at two of the buildings up to the present time. Both efflorescence and spalling have been stopped at one building for the past 2½ yr. by a very thorough waterproofing treatment.

The spalling in this group was in thin layers, frequently of large size, and this action progressed, layer after layer, until in the more advanced stages, the whole face of the brick has spalled off to the depth of ¼ in. The spalling has occurred only in the areas of abundant efflorescence. All of the noticeable growth of spalling in this group also has occurred in the months of April, May and June. Each year the condition has increased during these months and then remained apparently quiescent for the rest of the year.

When efflorescence was abundant during these periods of growth, heavy deposits of efflorescent salts almost invariably have been found back of the loosened layers of brick that were ready to spall off. I have made no careful observations of conditions obtaining

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[3] L. A. Palmer, "Cause and Prevention of Kiln and Dry-House Scum and of Efflorescence on Face-Brick Walls," *Technologic Paper No. 370*, Nat. Bureau Standards (1928).

during the spalling of common brick, but have frequently noted a similar bed of efflorescence under loose spalls when removed.

It is known that abundant efflorescence has recurred on many brick surfaces for years without causing any spalling, but if efflorescence is proved to be a hazard to the structure of certain kinds of brick, then the wick test of Messrs. McBurney and Parsons will assume added significance. I hope that the investigation of efflorescent salts in brick will be continued until their influence on spalling and disintegration is definitely known.

MR. C. H. CARMICHAEL.[4]—I should like to ask Mr. McBurney why he classifies two grades between none and moderate in his standardization.

MR. J. W. MCBURNEY.[5]—In answer to Mr. Carmichael's question, the insertion of two grades between "none" and "moderate" was for psychological reasons. "None" would be interpreted as "zero." If there were no intermediate grades between "none" and "moderate" the tendency would be to report very slight and unobjectionable amounts of efflorescence as "moderate" since obviously the efflorescence is not "none."

Mr. Palmer's discussion is very much appreciated since he has clarified several points which warrant more attention and emphasis than the authors gave to them. His comment on "cryptoflorescence" is an example of such a clarification.

It is of interest to note that brick No. 18 gave slight to moderate efflorescence in the 5-day wick test and slight efflorescence in the panel test, all of which was the green yellow scumming referred to by Mr. Palmer. This green yellow scumming is characteristic of bricks from the locality, Baltimore, Md., supplying brick No. 18. That Baltimore in years past was the seat of a considerable commercial production of chromium ores would appear to explain this rather unusual type of scumming.

Mr. Palmer very properly calls attention to mortars as sources of soluble salts. At the same time that the series of panels discussed in the present paper was built, another series, using a non-effloresing brick (No. 4), was constructed with a series of mortars ranging from 1:3 lime-sand by volume to 1:0.2:3 cement-lime-sand by volume. Panels built using 1:3 lime-sand and 1:2:9 cement-lime-sand by volume and with "protections" 1 and 3 gave considerable scumming on their surfaces. This scumming was caused by calcium hydroxide carried to the surface and there carbonated. Wilson in 1928[6] noted this type of efflorescence and explained it as caused by a considerable excess of water moving into the wall and thereby transporting calcium hydroxide to the wall surface. In commercial construction, with a reasonable degree of protection this does not occur.

With reference to Mr. Connor's discussion, it should be remarked that workers at the Building Research Station at Watford, England, are in complete agreement with him, judging by their published papers. English investigators apparently pay slight attention to frost action as an agent of disintegration but do regard the action of salts as important. Differences in climate may explain this difference in emphasis. It is probable that in the United States we have not paid the attention to the disintegrating effect of salt action that the subject warrants. We hope that some work will be done in that field.

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[4] Manager, Atlantic Brick Co., Watertown, Mass.

[5] Senior Technologist, National Bureau of Standards, Washington, D. C.

[6] Hewitt Wilson "Progress Report on the Efflorescence and Scumming of Mortar Materials," *Journal*, Am. Ceramic Soc., Vol. 11, No. 1, pp. 1-31 (1928).

SYMPOSIUM ON SIGNIFICANCE OF TESTS OF COAL

INTRODUCTION

> In order that the facts obtained by observation and experiment may be capable of being used in furtherance of our exact and solid knowledge, they must be apprehended and analyzed according to some conceptions which, applied for this purpose, give distinct and definite results, such as can be steadily taken hold of, and reasoned from.
>
> Whewell

Coal is a complex mixture of organic and inorganic materials. The chemical identity of only a few of these components is known. That the mixture must be variable in composition is evidenced by the wide variance of the properties of different coals. Because of the growing importance of coal in our civilization during the past century, as well as because of man's normal curiosity about this natural material which is "all things to all men," many methods of testing it have been proposed and tried through the years. Some of these have withstood the test of time and have become "standardized." Practically all of them are empirical, by which is meant that they do not, as a rule, measure properties of the sample but rather behavior under fixed conditions.

Interpretation is of utmost importance in all experimentation. It is vital in the case of tests of the nature of those used in fuel technology. The results of the tests are signs; what do these sign show? In other words, what do the tests signify? What is their meaning from the standpoint of the utilization of coal? Experience and judgment play important roles in the interpretation of the results of such tests. This symposium was arranged in order to provide an opportunity to a group of men of wide experience and mature judgment for exchange of ideas and complete discussion of the meaning of the results of tests of coal.

The papers and discussion have now been assembled in one place. The ideas and experiences of many technologists have been collated and are here presented for the benefit of all who care to use them. They are not beyond criticism. This is not the last word on the subject; in fact, study of the papers and discussion confirms the wisdom of Berzelius, who said, "Unfortunately, the certainty of our knowledge is at so low a level that all we can do is to follow along the lines of greatest probability."

Much is still to be done and the Symposium Committee sincerely hopes that this pamphlet will stimulate further study and discussion in this very practical field of technology.

Symposium Committee:
A. W. Gauger, *Chairman*
G. B. Gould
J. B. Morrow
Alden H. Emery

INTERPRETATION OF LABORATORY COAL TESTS—PROXIMATE ANALYSIS AND CALORIFIC VALUE

By G. B. Gould[1]

A discussion of the interpretation of laboratory tests of coal is hedged about by a number of inherent limitations, which must be explained at the outset.

There is great diversity in the chemical composition of the coal substance and in its mineral impurities and correspondingly in its physical and chemical properties. The only comprehensible approach to an understanding of such a material is to try to determine the significance of variations in each of its many properties, one by one. But the behavior of coal from a given mine is dependent upon the combination of all of its properties. Because of the conditions of geologic formation and the organic nature of the material itself, coals vary from one another by imperceptible gradations of the several properties, and the number of combinations is infinite. Some of these properties vary in a known relation to each other, while others vary quite independently. In practice, a variation in one of two independently varying properties may either accentuate the effect of the other property or cancel it.

As a practical matter, we are always dealing with a coal having a certain combination of all the known, and probably some unknown, properties. Nearly all interpretations of laboratory coal tests are relative. It is almost always a matter of the performance or the value of one coal as compared with another. Seldom do two coals differ from each other in respect to only one of their properties. Therefore, the interpretation of laboratory tests of coal is, in the present state of our knowledge, more in the nature of an art than a science, and is dependent, to a large extent, upon the experience of the individual.

The interpretation of laboratory tests of coal is rendered still more difficult by the variety of conditions to which coal is subjected in use. Coal is burned in a solid fuel bed supported by a grate; it is burned in pulverized form in suspension; and it is burned partly in suspension and partly on a grate. It is fired either by hand or mechanically. Mechanical firing may be of the underfeed or overfeed type. Fuel may be introduced at the front of a furnace moving to the rear; it may be introduced at the sides, progressing toward the center; it may be introduced at the center and progress toward the sides. The grate may be either inclined or flat. The fuel bed may be advanced by a combination of mechanical agitation and gravity, or transported without agitation on a traveling grate.

The ratio of boiler heating surface to grate area varies among plants from as low as 30:1 to 80:1, or even higher, in exceptional cases.

The rate at which coal is burned may be anywhere from less than 20 lb. per sq. ft. of grate per hour to as much as 100 lb. per sq. ft. The rate of burning may be nearly constant throughout the day in one plant, and in another be subject to frequent and substantial alteration. There is no uniformity in

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[1] President, Fuel Engineering Company of New York, New York City.

the size of furnaces related to the rate at which coal is burned in them, and they vary greatly in the amount of water-cooling surface exposed to the fire. These variables affect the maximum temperature to which the coal is subjected, and the rate of heating, both prior to and after it is ignited. Each plant, therefore, has its own individual combination of conditions which affect the behavior of coal in use.

The interpretation of laboratory coal tests is a problem of relating different combinations of properties of coal to a great variety of combinations of conditions of use. With an almost infinite number of possible combinations on each side of the equation, it is more difficult and seemingly more inconclusive to discuss the subject in general terms, than it is to deal with an actual case in practice.

When applied to a given case, one is dealing with one ascertainable combination of conditions of use. Starting with this, it is possible to simplify the problem with some generalizations about the properties of coal, which are valid and applicable to that set of conditions. It becomes possible to establish for each plant the significance of laboratory tests applicable to its own equipment and operating conditions.

In fact, a logical and rational approach to the solution of the problem would be to interpret the conditions of use in terms of coal properties, instead of attempting to interpret the laboratory tests themselves. But under present conditions that is possible only for an individual plant. A report of the Joint Committee on Fuel Values of the American Institute of Mining and Metallurgical Engineers and The American Society of Mechanical Engineers[2] contained the following paragraphs:

> (3) *Relative Effect of Properties of Coal Dependent upon Plant Characteristics.*—The effect of variations in any one or combination of physical and chemical properties of coal is likely to be relatively small within limits determined by the physical characteristics of each plant. As these limits are approached or exceeded the adverse effect becomes rapidly more pronounced. For this reason, the relation of the properties of coal to the capacity at which a plant must be operated is often of paramount importance, and in general is of greater importance in selecting coal for existing plants than are variations in efficiency or operating cost.
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> (4) *Plant Characteristics of Major Importance.*—The temperature of the fuel bed and furnace, and the rate at which coal must be burned per square foot of grate or per cubic foot of furnace constitute the principal factors determining the relative importance of the properties of coal required for satisfactory performance. Both in design of new steam-generating units and in the selection of coal for existing plants, the following plant characteristics should be given particular attention:
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> Furnace volume and design.
> Grate area.
> Water-cooling of walls and amount of radiant heating surface.
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> Any deficiency in furnace volume or grate area, or in equipment for conveying, crushing, pulverizing, or feeding of fuel, tends to accentuate the importance of the chemical and physical properties of coal.

And among the recommendations of the committee is the following:

> Investigation and discussion of steam-plant performance and data should give particular attention to the plant characteristics to which attention has been especially directed in (4) [quoted above] and to as complete description of the coal in use as possible, to the end that more critical data will be accumulated, which will relate fuel-bed and furnace conditions to specific combinations of chemical and physical properties of coal.

This kind of data is still, for the most

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[2] This report was filed with both societies, and printed in *Mining and Metallurgy*, July, 1936.

part, scattered, fragmentary, and unorganized. The interpretation of laboratory tests, under these conditions, must be in the nature of rather broad generalizations. Our knowledge of the chemical and physical properties of coal, imperfect as it is, is greater than the knowledge and understanding of the conditions of the use of coal.

The several properties of coal may singly or in combination affect either (1) the efficiency of the plant, (2) its operating cost, or (3) its maximum output; or any combination of them.

CALORIFIC VALUE

Of all the laboratory tests applied to coal, the calorimetric test is the only one which provides a measure of its intrinsic value as a fuel. The result is expressed in the United States and in Great Britain as "British thermal units per pound," and in countries using the metric system as "calories per gram."

The British thermal unit (B.t.u.) is the amount of heat required to raise 1 lb. of water 1 deg. Fahr.[3]

The B.t.u. per pound, in addition to being the only quantitative measure of the energy value of coal as a fuel, has a relation also to the operating cost and maximum steam output of a plant. Variations in B.t.u. ordinarily encountered in a given plant are of primary importance from the standpoint of the total energy received and usually only of secondary importance for their effect on operating cost or capacity. This varies greatly among plants, however, for in a large steam station, which is operating part or all of its steam generating units at maximum capacity, coal having a lower B.t.u. value might necessitate operating more units, or result in failure to meet the maximum demand. The great majority of industrial plants have a margin of unused capacity which is greater than ordinary variations in B.t.u. Failure to carry the load is more often due to the effects of some of the other properties of the fuel.

As between two coals differing in B.t.u. value, the one having the lower B.t.u. will generally tend to increase operating costs, because a greater quantity of material must be handled. The practical financial effect depends upon the plant, for reasons discussed at greater length under the heading "Percentage of Ash" below.

The number of B.t.u. per pound of coal is commonly expressed either as B.t.u. dry or B.t.u. "as received," the latter indicating the B.t.u. for the coal in its moist state. Similarly, phrases such as "as sampled," or "as fired," have the same significance.

"B.t.u. as received" represents the energy value of the coal as it is received at the laboratory, always containing a certain amount of moisture. If care has been taken to preserve the sample in an air-tight container, between the time the sample is taken and the time it is delivered to the laboratory, this is the figure which most nearly represents the B.t.u. value of the coal as it is used. Because of the instability of the moisture content, the significance of the "B.t.u. as received" is more particularly discussed under the heading of "Moisture" below.

The B.t.u. determination in the calorimeter is commonly made with a sample which has been air dried, the moisture content of which is reasonably stable. The B.t.u. value is then corrected for the moisture in the sample at the time the calorimetric determination is made to derive the "B.t.u. per pound

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[3] The British thermal unit is commonly defined as $\frac{1}{180}$ of the heat required to raise 1 lb. of water from 32 to 212 F. No official definition of the B.t.u. has been adopted by any professional or scientific body competent to do so. The B.t.u. is required for all calculations of the efficiency of steam-generating units, the rate of heat release, etc., for which reference should be made to the A.S.M.E. Boiler Test Codes.

dry."[4] Absolutely dry coal does not exist in actual use up to the time coal is fired. The "B.t.u. dry" is, however, a convenient term for comparison of values among coals of the same kind and size, in order to eliminate the influence of accidental variations in moisture which result from variations in weather conditions during shipment or storage.

It is desirable to understand, in a general way, the relation which exists between the B.t.u. and the rank of a coal, even though the B.t.u. per pound of "pure" coal, which is a theoretical abstraction, has no direct application in the interpretation of a coal analysis for practical purposes.

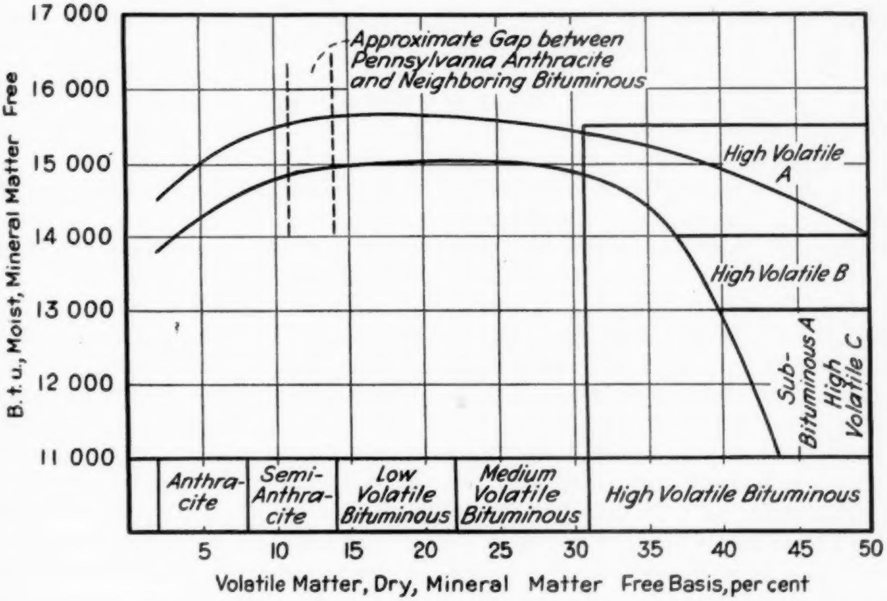

FIG. 1.—Classification of Coals of the United States by Rank.

Figure 1 is a simplified graphic generalization of the data shown in Figs. 1 and 2 of the report on "Classification Chart of Typical Coals of the United States," by Fieldner, Selvig and Frederic.[5]

It will be seen that the B.t.u. value, volatile matter, fixed carbon and moisture are all interrelated. Since the percentages of fixed carbon and volatile matter in pure coal are complements of each other, reference here is made only to the percentage of volatile matter.

The B.t.u. values plotted on the chart are for coal containing the natural bed moisture, which is considered as an inherent and inseparable constituent of the coal, but excluding by mathematical approximation[6] the mineral matter.

As the percentage of volatile matter rises, the B.t.u. per pound of pure coal first rises and then declines. Up to about 35 per cent volatile matter (dry,

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[4] The B.t.u. dry is derived by the following formula:

$$\frac{\text{B.t.u. as received} \times 100}{100 - \text{per cent of moisture}} = \text{B.t.u. dry}$$

[5] *Report of Investigations No. 3296*, U.S. Bureau of Mines, December, 1935.

[6] For approximation formula see Tentative Specifications for Classification of Coals by Rank (D 388 – 36 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 812 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 520.

mineral matter free basis), variations in the natural bed moisture are small, and the percentage of volatile matter is the primary indication of rank, and correspondingly of the range of B.t.u. per pound of pure coal. From about 35 per cent volatile matter upward, the amount of natural bed moisture increases substantially, becomes a significant indicator of rank, and correspondingly reduces the B.t.u. value. The reason the B.t.u. value is related to the percentage of volatile matter is that while the B.t.u. value per pound of fixed carbon remains constant, that of the volatile matter varies considerably according to its composition.

In general, the coal from a given mine has a characteristic B.t.u. value for the pure coal, but there are differences among the sizes, especially the very small sizes, from the same mine. This is due to the fact that the coal itself is not a homogeneous material, but a combination of materials which differ among themselves in hardness and other physical and chemical properties. The result is that the smaller sizes may contain a disproportionate share of the more friable material, notably fusain, which is relatively low in B.t.u. value.

From a geographical standpoint, the lowest percentage of volatile matter is found in the most easterly of our coal beds, and rises westward to the Mississippi River. Beyond the Mississippi, coals having a wide range of volatile matter are found in close geographical proximity, notably in Oklahoma, Arkansas, Colorado, and Washington. In the East, there is a gap between the semi-anthracite and the lowest volatile bituminous coal, from about 11 per cent volatile matter (dry, mineral matter free basis) to about 14 per cent. Otherwise the progression from low to high volatile is continuous.

The B.t.u. is a primary factor in coal valuation, for it is the measure of the total amount of energy in the coal. The proportion of this total energy which is recovered in a given plant under a given set of operating conditions, and the operating cost incident to the conversion are affected, favorably or unfavorably, by variations in the chemical and physical properties which are disclosed by other laboratory tests. In other words, the B.t.u. is the *only* positive measure of the value of coal as a fuel, while the other laboratory tests are negative indications, of varying degrees of importance, depending upon the conditions of use.

Moisture

Moisture must be considered both for its effect on plant performance and for its relation to other measurements and calculations.

Moisture is inherent in the coal structure, but it may be increased by seepage in the mine, and between the time the coal is removed from the seam and its use, moisture may be added in the process of cleaning, or by rainfall or snowfall while the coal is in transit or storage. Moisture may also be lost to the atmosphere, and in the case of excessive amounts, by drainage during transportation or storage. The moisture content of coal, therefore, may change in either direction at any time, depending upon the conditions to which the coal is subjected.

Moisture in coal affects the efficiency of a steam-generating unit directly, only to the slight extent of the heat content of the steam resulting from its evaporation, at the temperature of the flue gases at the exit of the steam-generating unit. This loss is approximately 0.1 per cent of efficiency for each 1 per cent of moisture in the coal.

High *surface* moisture, that is, moisture mechanically retained on the surface of the coal particles or between them, adversely affects the performance of some pulverizers. The effect is chiefly one of reducing the capacity of the pulverizer, and its importance in a given case depends upon whether or not the required output is close to the maximum capacity of the pulverizer.

While the precise effect is a matter of speculation, it is known that the addition of surface moisture to coking coals before firing has a distinctly beneficial effect on the structure of the fuel bed, particularly in the absence of mechanical agitation, as in the case of the chain grate. Observations in practice indicate higher efficiency resulting from the greater uniformity and porosity of the fuel bed, and an increase in the rate of ignition.

High surface moisture, on the other hand, may adversely affect the flow of coal in bunkers, spouts, and other conveying devices, depending upon their design, and because of freezing, accentuates difficulties in unloading or recovering coal from storage in sections where low temperatures are encountered.

The capacity of a coal to retain surface moisture is chiefly determined by the size of the pieces and the uniformity of size. For example, it is a matter of common observation that the amount of moisture which will be normally retained by the several commercial sizes of anthracite rises as the size diminishes. This kind of coal has a rather high order of size stability, is quite uniform in size, and in a well-prepared product contains not over 15 per cent of smaller sizes, with a minimum percentage of dust.

Bituminous coal, however, differs among producing areas and among mines in size stability, and the great bulk of it which is used in steam plants contains a relatively wide range of sizes. Much of this coal, known as slack or nut-and-slack, contains everything which passes a screen of given size. The capacity of such coal to retain surface moisture, because of the assortment of sizes, is greater, but it also offers greater resistance to the penetration of moisture while in transit or in storage. When such coal is rehandled during a heavy rain it may pick up a comparatively large amount of water, which it will then stubbornly retain during subsequent storage.

High surface moisture in combination with sulfur promotes corrosion of all metal surfaces with which the coal comes in contact during transportation or storage.

Because of the instability of moisture, the interpretation of the laboratory determination must take into consideration the time when the sample was taken and the handling of the sample up to the time it reaches the laboratory. If the percentage of moisture in the coal at the time of sampling has a significant relation to any subsequent calculation, the sample should be placed immediately in a sealed container.

When the weight of coal enters into a calculation, maximum accuracy requires a determination of the percentage of moisture present at the time of weighing. This is frequently impracticable or the error involved does not justify the practice, but the principle should be understood.

It is common commercial practice to ignore the percentage of moisture in the coal at the time railroad car weights are determined. Whatever the moisture content is at that time, it is billed to the buyer as coal.[7] The variation in moisture content among coals of similar kind and size is not usually of sufficient magnitude to justify the expense of its determination at the scales as a basis for relative valuation of two or more coals.

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[7] An exception to this is found in the arbitrary allowances for moisture in weighing anthracite at the mines.

The use of water-cleaning may add to the moisture content of a coal, but methods are in use to dewater the coal after cleaning. The buyer can judge of the percentage of moisture at railroad scales only by the moisture content found in the coal at the time of arrival. If comparison is to be made between two coals on the as-received basis, such a comparison should be based only on laboratory tests covering shipments over a long enough period to eliminate the vagaries of weather as a factor. There is an observable seasonal variation in the percentage of moisture in coal at the point of delivery, reaching a maximum in midwinter and the minimum in midsummer. Individual shipments will vary according to the weather during shipment.

Additions to or losses from the amount of moisture present at the time bill-of-lading weights are determined do not affect the validity of those weights for billing purposes, because these changes, while they increase or decrease the weight finally received, do not change the total energy value of the shipment.

If coal is paid for on the basis of weights at the time of delivery, as in the case of truck deliveries from a yard, the moisture can be more easily determined, but only the amount of moisture present at the time of weighing affects the total energy value paid for.

Coal is also weighed at, or just prior to, the time of firing, for the purpose of determining the performance of the steam generating unit. Calculation of efficiency, based on these weights, requires determination of the percentage of moisture present at the time of weighing. If a sample taken prior to or some time after the coal is weighed shows a lower percentage of moisture than the coal contained at the time of weighing, then when the results of the laboratory test are combined with the observed weight, the apparent total energy input will be greater than the actual energy input. And, of course, the converse is likewise true.

It is obvious that as moisture is increased or decreased, there will be a proportionate decrease or increase in the B.t.u.'s per pound as received, but there will be no change in the total number of B.t.u.'s.

This inherent instability in the moisture content of coal introduces a practically inescapable probable error in calculations of steam-plant performance of at least ± 1 per cent, and in many cases twice as much, unless the moisture is determined from samples taken and sealed at the time the coal is weighed to the boilers. Comparative figures for continuous operation of a given plant are not so seriously affected. The important thing to understand is that accuracy requires that sampling must be timed according to the use which is to be made of the resulting laboratory test, if the weight of the coal enters into the calculations.

PERCENTAGE OF ASH

Ash is the incombustible mineral material which remains after coal has been burned. It is not identical with the material commonly referred to as "ashes," which are removed from the furnace in commercial use, and which invariably contain varying proportions of unburned coal.

It is difficult to separate in one's mind the effect of ash in coal as a material to be handled, from its effect in diminishing the B.t.u. per pound of a coal as its percentage increases. Obviously, as the proportion of ash increases, the proportion of heat-producing material decreases, and the B.t.u. per pound of a given coal diminishes proportionately. The effect of lower or higher B.t.u. on plant performance should properly be considered under the heading of B.t.u., recognizing, of course, that a lower B.t.u. value is coincident with a higher percentage of ash in coal of the same rank. It should be pointed out that of two bituminous coals having the same B.t.u. value, the coal of lower rank, in general, will have the lower percentage of ash. Or to put it another way, a high-rank bituminous coal may have a higher percentage of ash than a low-rank coal, and still equal it in B.t.u. value. The reason, of course, is that the high-rank bituminous coal has a higher B.t.u. value per pound of pure coal.

What follows has to do with the effect of ash as a material which must be handled before and after the combustion process, and which affects the process itself. These effects are numerous, and will vary in importance according to the type of plant and the conditions of its operation, and the range of ash percentage under consideration. They are well summarized in the report of the Joint Committee on Fuel Values of the American Institute of Mining and Metallurgical Engineers and The American Society of Mechanical Engineers as follows:

> *Ash.*—Adverse effect on efficiency is found chiefly in case of solid fuel beds, resulting from entrainment of carbon by the ash, but in all plants ash may affect efficiency by fouling of heating surfaces. High ash, owing to the additional bulk of material that must be handled to deliver a given number of B.t.u., reduces the capacity of all fuel- and ash-handling and fuel-burning equipment. (This general statement needs to be slightly modified when one of two coals has a substantially higher ash- and moisture-free B.t.u. value than the other, in which case the coal having the higher B.t.u. may have the same or slightly higher percentage of ash, but results in a smaller quantity of ash to be handled per unit of steam generated, at the same efficiency.) Capacity may also be reduced as a result of fouling of heating surfaces, or reducing available draft, or indirectly by reducing efficiency.
>
> Operating costs are also influenced by the percentage of ash, because of additional quantities of fuel and refuse to be handled and disposed of; erosion by fly ash of equipment between the furnace and the stack, increased cost of power and maintenance of pulverizers, and increased cost of cleaning.

How widely the effect of the percentage of ash may vary among plants, from the standpoint of the amount of material to be handled, can be seen by an illustration. A small plant requires the services of one man to handle both coal and ashes, and he has plenty of time. A larger quantity of ash to handle does not increase the cost of ash handling, until it reaches the point where additional labor is required. A smaller quantity does not reduce the handling costs, because no reduction in labor is possible. In addition, this plant may be so located that its ashes can be sold, or possibly used for fill which otherwise would have to be purchased. On the other hand, a larger plant has more chance of varying its labor force according to the amount of material to be handled, and such mechanical ash-handling equipment as it has may be close to its capacity limit, with a low-ash coal. In addition, the plant may be so located that the ashes have to be carted a considerable distance to be finally disposed of. In such a plant, the operating costs will be directly affected to a noticeable extent by an increase or decrease in the percentage of ash.

The percentage of ash has a much more pronounced relative effect on the amount of refuse to be handled than upon the amount of coal which must be used. For example, if one coal contains 5 per cent of ash, and another coal 10 per cent, the use of the latter coal will involve the handling and disposal of more than twice as much refuse, while the amount of coal required will be only 5 per cent greater, assuming that other factors remain constant. In very small plants, there is the tendency sometimes to jump to the conclusion that twice as much ash to remove means twice as much coal consumed. This is not true if the increase is caused solely by a higher percentage of ash in the coal.

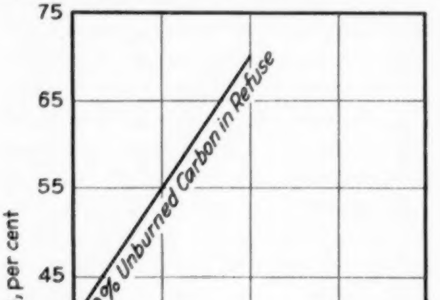

FIG. 2.—Relation of Ash and Coal to Loss in Refuse. Low-volatile bituminous coal.

Likewise, the effect in the fuel bed itself is not proportional to the amount of coal being consumed in a given time, but more nearly to the amount of incombustible material present. As between a 5 per cent ash coal and one having 10 per cent ash, the amount of incombustible material present on the grate will be at least double in the latter case. If clinkers are being formed, or likely to be formed in a given case, and if the fusing temperature of the ash of the two coals is the same and other factors remain constant, it is obviously an important fact that with the 10 per cent ash coal there is present at least twice as much material out of which clinkers may be formed.

There is one adverse effect of ash on the efficiency of a steam-generating unit which appears to be universal for all plants burning coal in a solid fuel bed. That is the inevitable entrainment of some unburned coal. The amount of this entrainment is determined, to a large extent, by the kind of grates, the type of stoker, and the rate of combustion, but in a given case, these factors remaining constant, the loss due to unburned coal in the refuse is probably proportional to the percentage of ash. I say "probably" because the variation in this loss due to this cause is of such a low order of magnitude that it is less than the probable error in determining the efficiency of a steam-generating unit.

Data derived from "A Study of Four Hundred Steaming Tests"[8] conducted by the U. S. Geological Survey in 1904 to 1906 indicates that the drop in efficiency of boiler and furnace is approximately 0.20 per cent for each 1.0 per cent increase in percentage of ash. These tests were all conducted with hand-fired boilers, operating at what would be considered low rates of combustion today. Somewhat similar conclusions were reached from a more recent series of tests on chain-grate stokers, conducted by B. M. Thornton, of the Imperial Chemical Industries, Ltd., in England.

Referring to Thornton's tests, E. S. Grumell said:[9]

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[8] Lester P. Breckenridge, "A Study of Four Hundred Steaming Tests," *Bulletin 325*, U. S. Geological Survey (1904–1906).

[9] E. S. Grumell, "The Evaluation of Fuel from the Consumers' Viewpoint," *Journal*, Inst. of Fuel, August 1932, p. 361.

Owing to some special features, Thornton's figures are on the low side and it would be safer to assume that without certain precautions the efficiency decreases by one-third of 1 per cent for each increase of 1 per cent in ash content. In certain cases, for instance, when the loss of carbon in ashes is not carefully controlled, and where the coal is of such poor quality that appreciably greater draught is required to maintain output, the effect on the efficiency may be somewhat greater, but the fact that results of independent studies of the effect of ash on efficiency give such closely agreeing results indicates that, as a generalisation, it may be accepted without running the risk of serious errors.

Because of the small variation in efficiency which is involved, a clearer and possibly a more accurate understanding of the relation between percentage of ash and efficiency can be obtained by a process of deduction. Within the range of percentage of ash ordinarily encountered in coals of merchantable quality, the percentage of combustible in the refuse for a given plant and rate of burning seems to be practically constant, regardless of the percentage of ash; or if it increases with the percentage of ash, it is by an amount which is not significant.

Taking a percentage of unburned combustible in the refuse which is found to be characteristic of a given plant, it is possible to calculate the resulting heat loss.[10] An increase of 1 per cent in this loss reduces the efficiency by 1 per cent. Such a calculation is represented graphically in Fig. 2 for refuse containing in one case 20 per cent of unburned combustible, and in the other case 40 per cent, for a typical high-rank low-volatile eastern bituminous coal. The rate of increase in the loss due to unburned combustible, when the percentage in the refuse is 20 per cent, will be seen to be approximately 0.3 per cent for each 1.0 per cent of ash. When the percentage of unburned combustible in the refuse is 40 per cent, the rate of increase in the loss due to unburned combustible is approximately 0.75 per cent for each 1.0 per cent of ash. This is the theoretical relationship which can be expected in practice up to the point where the percentage of ash becomes so high that it seriously interferes with the distribution of air throughout the fuel bed. Typical performance will usually fall somewhere between these two limits. The rates indicated by these calculations are somewhat higher than the deductions made from the observed data in the two series of tests, mentioned above, but in both series of tests there was the possibility of a counterbalancing effect of other factors.

This relationship refers only to the effect of ash through entrainment of unburned fuel. It does not mean that of two coals differing in percentage of ash, as well as in other properties, the efficiency of the steam generating unit will necessarily be lower with the coal having the higher percentage of ash. It is not uncommon in practice that a coal having a higher percentage of ash more than compensates for a higher loss due to unburned combustible in the refuse, by other properties such as size or coking characteristics, which are favorable to a lower dry flue gas loss.

Size and coking characteristics are beyond the scope of this paper but are mentioned in this connection because of their possibly modifying effect and as an illustration of the general fact, stated earlier, that in actual practice the interpretation of a coal analysis is a matter of judging the effect of a certain combination of all the properties of coal.

PERCENTAGE OF VOLATILE MATTER AND FIXED CARBON

Small differences in the percentage of volatile matter are usually of little significance in practice, and such rela-

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[10] For this formula, see A.S.M.E. Boiler Test Code.

tion as there is to plant requirements and plant performance is strictly relative to the particular plant.

Pure coal (moisture and mineral matter free) is empirically divided between volatile matter and fixed carbon. The percentage of volatile matter, which comprises one of the items in the proximate analysis, is obtained by heating the coal at 950 C. for 7 min.[11]

That portion of the coal which is driven off as a gas by this treatment is considered the volatile matter. The remaining material, exclusive of moisture and mineral matter, is considered to be the fixed carbon. This is a laboratory method for distinguishing between that part of the coal which is burned as a gas, and that part which is burned as a solid.

As a practical matter, coal constitutes a mixture of fuels, each one having distinct chemical and physical properties. The proportions of gaseous and solid fuel vary over a wide range, from anthracite, which contains the lowest percentage of volatile matter, through the various ranks of bituminous coal to lignite and finally peat. All methods of burning coal constitute a two-stage process, more readily recognized when coal is burned on a grate, in which case the volatile matter is expelled and burned rapidly (if at all) above the fuel bed, while the remaining solid fuel is burned more slowly, usually in the form of coke, except in the case of anthracite.

"When the volatile matter in coal is not properly and intimately mixed with oxygen, the hydrocarbon molecule is 'cracked' into carbon and hydrogen, the latter burning to water while the former passes through the boiler and up the stack. This form of solid carbon is so finely divided that it becomes classified definitely as a 'smoke.' This is 'carbon black' and curiously enough it is practically non-combustible, except under favorable circumstances, such as high temperature and an atmosphere rich in oxygen. Most of us are acquainted with this form of carbon from its collection in the cylinders and on the valves of our automobiles."[12]

Such laboratory experiments as have been reported indicate that when coals are burned under controlled laboratory conditions, the amount of smoke is proportional to the percentage of volatile matter.[13]

The method of burning coal on a grate is obviously better adapted to the efficient combustion of the solid portion, which is mechanically supported for a sufficient time to effect almost complete combustion, under conditions which permit the regulation of the air supply to the rate of burning and the thickness of the fuel bed. The volatile matter, however, is released in an atmosphere deficient in oxygen, which needs to be supplemented by the introduction of air above the fuel bed. Its combustion must be effected quickly, or not at all; consequently, conditions which favor its combustion include a furnace large enough to permit thorough mixing with air and time enough for combustion before it reaches the colder parts of the furnace and boiler passes, provision for the introduction of air in quantities and at the places where it is needed, and a relatively high furnace temperature. None of these conditions, except the supply of air, is necessarily a requisite for burning the solid portion of the fuel.

It follows, therefore, that in the gradual evolution of the coal-fired boiler, many furnaces were designed and in-

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[11] See Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 – 33), 1936 Book of A.S.T.M. Standards, Part II, p. 387. For sub-bituminous lignite and peat the prescribed time of heating is 6 min.

[12] Douglas Henderson, "Of These Two Which Is Smoke," *Power*, January 12, 1932.

[13] Illinois Geological Survey—*RI 41*, Part II.

stalled and are still in service, which provide insufficient space and inadequate means of introducing properly distributed over-fire air. Many of these plants are also operated at low-combustion rates with correspondingly low furnace temperatures, with the result that a considerable portion of the volatile matter escapes unburned. This causes some loss of heat which would otherwise have been extracted, and the emission of smoke. In such plants, the percentage of volatile matter is a consideration of major importance in coal selection, the degree of importance depending upon the design and operating conditions of the particular plant, and the desirability of avoiding smoke.

The proportion of total heat value contributed by the volatile matter in bituminous coals will range from about 25 to 40 per cent. Since the heat produced by the volatile matter is quickly available, the higher volatile coals are generally considered more "flashy" and better adapted to meeting sudden increases in load. Other properties of coal, however, such as the coking characteristics, as they affect the porosity of the fuel bed, the size of the coal, and the ignition temperature of the solid part of the fuel, also affect this adaptability to sudden demand.

When coal is burned in pulverized form, in suspension, the volatile matter is released more rapidly in an atmosphere still rich in oxygen, mixes with it quickly by diffusion, and the furnace temperature is higher than is found in a great majority of hand-fired or stoker-fired furnaces. The combination of conditions is very well adapted to the combustion of the volatile matter. Pulverized coal burning plants, therefore, do not ordinarily produce true smoke, such as is produced by other kinds of plants, but rather a dust consisting of particles which, though they appear very small to the eye, are actually very much larger in size than those produced from the volatile matter. "When the particles are coarse and, on account of their size, fall freely under gravity, the condition is called a 'dust.' If the particles are small enough to fall slowly at a constant velocity, it is known as a 'cloud.' When, however, the particles are so extremely small that they show no signs of settling, but are driven about by the bombardment of the gas molecules, a condition exists called a 'smoke'."[14]

In a pulverized-coal furnace the solid portion of the coal, now in the form of finely divided particles which are naturally slower to burn, must be permitted sufficient time to burn, and must be kept in contact with a steadily renewing supply of air in the presence of a high temperature, or it will escape unburned. Thus, in the pulverized coal burning plant, the more difficult stage of the two-stage process is to burn completely the solid portion of coal. The heat rapidly released in the combustion of the volatile matter aids in the ignition of the solid, or fixed carbon.

Consequently, viewed solely from the standpoint of the percentage of volatile matter, those coals having the higher percentage of volatile matter are better adapted to burning in pulverized form.[15] Other properties of coal which vary independently of the volatile, such as grindability, percentage of moisture, and the coking or non-coking nature of the coal, also affect its adaptibility to pulverizer service, and in particular cases may substantially modify the broad generalization in favor of higher volatile.

The percentage of volatile matter

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[14] W. E. Gibbs, "Some Factors Affecting the Problem of Smoke Prevention," *Journal*, Inst. of Fuel, June, 1930.

[15] For more complete discussion of this point, see two articles by Douglas Henderson, "How Pulverized Coal Burns, as Shown by the Microscope," *Power*, July 28, 1931; and "Diffusion as a Factor in Burning Pulverized Coal," *Power*, February 17, 1931.

serves a very useful purpose, because in combination with other determinations it identifies a coal with a certain section of the coal fields, a group of mines, a seam or its rank.[16]

In ordinary commercial practice the percentage of fixed carbon carries no direct significance of its own. Being essentially the complement of the percentage of volatile matter, the mental concept of the nature of a coal, derived from the other items of the analysis, is not augmented by the percentage of fixed carbon, and no conclusion arrived at without it would be modified by its inclusion.

UNIFORMITY

A discussion of the interpretation of coal analyses would be incomplete without some reference to the importance of uniformity of the properties of coal produced by a particular mine, or group of mines, contributing to a single commercial product.

If any of the properties of coal are of substantial importance to the satisfactory performance of a given plant, the degree of uniformity of those properties is, in itself, a characteristic of the fuel which must be considered. Excessive variability in any of these properties tends to reduce the efficiency of a plant, because of the necessity for frequent adjustment of combustion conditions. The maximum assured capacity of a plant can be no greater than that which corresponds to the greatest adverse deviation of any one of those properties which happen to be of primary importance in that plant.

TOLERANCES IN THE INTERPRETATION OF LABORATORY COAL TESTS

The interpretation of laboratory coal tests requires an understanding of the variations which occur, and their causes. This is particularly important in all commercial transactions and for engineering calculations which are based on laboratory tests.

Coal is a heterogeneous material, a mixture of pure coal and various mineral impurities. Some of these impurities are intimately associated with the coal, and some of them are free impurities which become mixed with the coal in the process of mining. The free impurities are seldom distributed uniformly throughout any particular lot of coal. The quantity of them in the product of any mine varies from day to day, from week to week, or from month to month. Individual coals differ in the amount of the impurities, in the proportions of inherent and free mineral matter, and in the uniformity of their distribution.

In sampling and testing such material, it is inevitable that there will be variations among the results, from car to car, from day to day, and even among samples taken from the same lot. Some of these variations arise from the difficulties of sampling, and some from the actual variations in the material itself.

Extensive statistical studies of the results of laboratory coal tests, both in this country and in England,[17] show that each coal has a characteristic deviation from its own average which is determined by its physical properties and by the conditions of its production and preparation for market. These factors can be summarized briefly, as follows:

1. Percentage of ash in the coal,
2. Size,

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[16] As to rank, see above under B.t.u.

[17] See E. S. Grumell and A. C. Dunningham, "Sampling of Small Fuel up to 3 in.," *No. 403*, British Engineering Standards Assn., and J. B. Morrow and C. P. Proctor, "Variables in Coal Sampling," *Technical Publication No. 645*, Am. Inst. Mining and Metallurgical Engrs. Most of the statements in this section, based on statistical studies of tests taken from the records of the Fuel Engineering Company of New York are in substantial agreement with these two reports, and repeated detailed references are omitted.

3. Ratio of free impurities to total impurities,
4. Nature of the free impurities,
5. Methods of mining, loading, and preparation,
6. Shipment from two or more mines as a single commercial product.

There seems to be what can be called a normal relationship between the percentage of ash and the deviations from the average of any particular coal, which applies to the majority of American bituminous coals in the small sizes ordinarily used for steam generation. Any substantial divergence from this norm can usually be traced to the effect of some one of the other factors. While not as much work has been done on the variations in other laboratory determinations, it is probable that the statistical relationship will be similar to those found for the percentage of ash, which represents the principal impurity in coal.

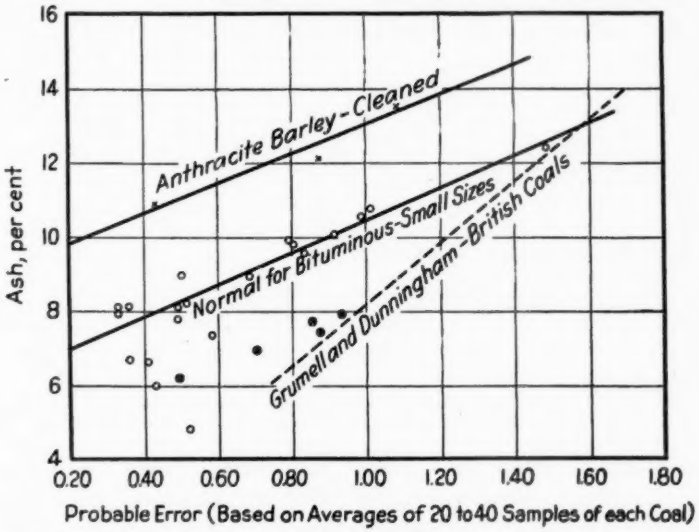

FIG. 3.—Relation of Probable Error of Individual Samples to Percentage of Ash. (Fuel Engineering Co. tests.)

Figure 3 illustrates the effect of some of these factors, showing the relation between the percentage of ash, and the probable error for each of 25 bituminous coals, and 3 anthracites. (The probable error is the deviation from the average of each coal, above and below which an equal number of tests may be expected to fall.[18])

The curve marked "Normal for Bituminous—Small Sizes," is based on the deviation of 20 to 40 individual samples from their respective averages, taken from the records of the Fuel Engineering Company of New York. The points represent what may be termed the characteristic error of each of the 25 bituminous coals. These coals were selected at random from the more important coal-producing areas in Pennsylvania, West Virginia, Kentucky, and Illinois. Five points are distinctively designated. Four of these are for coals which are

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[18] The word "error" has an unfortunate and misleading connotation in this connection, at least to the layman, to whom the word usually implies a preventable inaccuracy. It is a deviation from the average, due to the nature of the material, which is not preventable, and apparently capable of only slight modification within the limits of sampling methods which are practicable in ordinary commercial usage and which are economically appropriate to the value of the commodity. There is no way of telling how much of it is, strictly speaking, "error," and how much is a reflection of actual variations in the material itself. The characteristic error of a coal is one of its properties with respect to which it differs from other coals, and represents a factor which needs to be taken into consideration in the interpretation of laboratory tests.

produced from two or more mines, but in each case shipped as a single commercial product. The fifth, which falls at the insertion of 6.9 per cent and a probable error of 0.70, represents a 1 by 2-in. nut coal of hard structure, from eastern Kentucky. The divergence of the probable error from the norm, in the case of the four coals shipped from groups of mines, is undoubtedly due to that fact, and cannot be reduced by sampling procedure. The error for the 1 by 2-in. nut coal is probably due to a failure to reduce the size of the coal sufficiently as the samples were reduced, but it may also reflect an uneven distribution of free impurities, which is more likely to occur in coal of this size and structure.

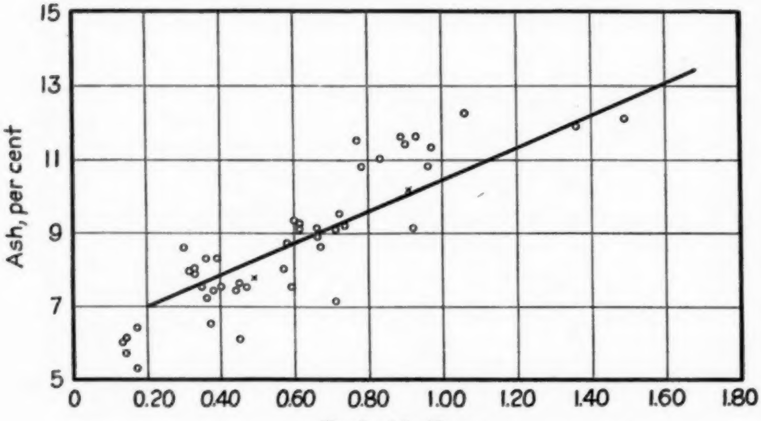

FIG. 4.—Relation of Probable Error to Percentage of Ash.

Car samples, bituminous, 2 in., and smaller. Data from paper from Morrow and Proctor.

Similar points are plotted for the product of three collieries producing mechanically cleaned No. 3 buckwheat (barley) anthracite. While the number of points plotted is not enough to be conclusive, they suggest a distinctive relationship between percentage of ash and the probable error, indicating a definitely lower error than is found for bituminous coal having the same percentage of ash. This is to be expected because of the combination of a small and uniform size and the exclusion of a large part of the free impurities by cleaning.

The samples on which the Fuel Engineering Company records are based were taken, for the most part, by ordinary boiler-room employees in a great number of industrial and public utility plants. These tests represent the result of instructed, but not supervised, sampling by men who have no special knowledge of the theory of sampling and, most of them, lacking in any scientific training. The value of these data lies in their reflection of the deviations to be expected from ordinary commercial sampling.

The relation between percentage of ash and probable error for British coals, found by Grumell and Dunningham, is also shown in Fig. 3 for reference. I am informed that British bituminous coals generally contain a larger proportion of the ash in the form of free impurities, which would account for the somewhat larger deviation which they find for the lower ash coals. Their curve intersects ours in the neighborhood of 13 per cent ash, at which point, it may be assumed, the proportion of free impurities in our coals approximates that of the British coals.

Figure 4 has been constructed from data in Morrow and Proctor's paper, for minus 2-inch coal and smaller. There is this difference between their data and those on which Fig. 3 was based: the tests reported by Morrow and Proctor were all for samples taken by trained men, under carefully supervised and standardized conditions, and represent a small number of mines in one section of the coal fields.

The curve, representing the normal relation between percentage of ash and the probable error in Fig. 3, has been repeated in Fig. 4, and it is interesting to note the close relationship between the two sets of data, considering the difference in the way the samples were taken. Two points on Fig. 3 represent coals which are also included in Morrow and Proctor's data, and these are repeated on Fig. 4 for reference, shown as x. The group of points falling between 10.5 and 12.0 ash, suggest that a considerable reduction in the probable error for the high ash coals resulted from the more expert sampling, but below 10 per cent ash a reduction in the characteristic error does not seem so easy to obtain.

The question naturally arises as to whether the probable error, which appears to be typical of present-day commercial practice, can be materially reduced by better methods and more rigid adherence to them. It is beyond the scope of the present paper to discuss sampling methods, but judging from the data now available, it is the author's opinion that for the great majority of bituminous coals having no more than 8 per cent ash, in the slack and nut-slack sizes, no significant reduction in the probable error can be obtained by any method which would be economically practicable to employ in the typical steam plant.

While gross violation of the elementary principles of sampling will undoubtedly increase the magnitude of the deviations from the average, the price of reducing them below that encountered in reasonably careful industrial practice today is probably higher than the consumer can afford.

Extra care should be taken, however, for coals higher in ash, or for the larger screened sizes (nut, stove, or egg), especially in the reduction of the size of the coal as the size of the sample is reduced.

The present A.S.T.M. Standards (D 21 – 16)[19] for the reduction of a sample are as follows:

WEIGHT OF SAMPLE TO BE DIVIDED, LB.	LARGEST SIZE OF COAL AND IMPURITIES ALLOWABLE IN SAMPLE BEFORE DIVISION, IN.
1000 or over......	1
500............	3/4
250............	1/2
125............	3/8
60............	1/4
30............	3/16 or to pass a 4760-μ (No. 4) sieve

Some coals, not necessarily high in percentage of ash, have a considerable proportion of free impurities, in the form of hard rock, which does not break down in the process of loading and handling. In such cases, the ash tends to be unevenly distributed, and consequently even in the smaller sizes special care should be taken to crush the sample properly.

Under these circumstances, it is important for the consumer and producer alike to recognize the magnitude of normal deviations in commercial practice (as opposed to theoretical standards of accuracy), to understand the cause of the deviations, and to rely more

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[19] Standard Method of Sampling Coal (D 21 – 16), 1936 Book of A.S.T.M. Standards, Part II, p. 382.

upon the average of a series of tests. Very broadly generalized, this means that for bituminous coals of small size and having not more than 8 per cent ash, the probable error of individual samples to be expected is from ±0.30 to ±0.50 per cent, and for coals having from 8 to 10 per cent ash, between ±0.50 and ±0.90 per cent.

The distribution of deviations from the average, in the case of coal, conforms quite closely to the theoretical distribution,[20] as shown in Table I. The distribution of the deviations from the average in terms of multiples of the probable error (*r*) for the 640 samples of bituminous coal used in the construction of Fig. 3, compared with the theoretical distribution, is as follows:

TABLE I.—DEVIATIONS OF COAL FROM THE AVERAGE IN MULTIPLES OF PROBABLE ERROR.

	Percentage of Tests	
	Actual	Theoretical
r	50.9	50.0
2*r*	84.8	82.3
3*r*	96.3	95.7
4*r*	98.6	99.3
5*r*	99.8	99.7
6*r*	100.0	100.0

Therefore, when the probable error characteristic of a given coal is known, the frequency of the larger deviations can be predicted. If an 8 per cent ash coal has a probable error of 0.50 per cent of ash, one half of all tests may be expected to fall between 7.5 and 8.5 per cent ash, approximately another one third of the tests between 7.0 and 7.5 per cent, or between 8.5 and 9.0 per cent, and about 13 per cent of the tests will be between 6.5 and 7.0 per cent, or between 9.0 and 9.5 per cent. The remaining 4 per cent of the tests will show even greater deviations.

These deviations from the average include the small unpreventable errors in analysis, errors due to sampling, and variations in the quality of different lots of the same coal. Characteristics of a coal which increase the range of variation of its quality also increase the chance of error in sampling. There is, therefore, a cumulative effect, starting with the nature of the individual coal, which is reflected in the final result. We are dealing here with the use of laboratory tests for estimating the value of a coal as an identified, but somewhat variable, commercial product, of which the buyer will ultimately receive a number of shipments over a period of time. It is important to make a clear distinction between this use of laboratory tests, which represents by far their most significant commercial application, and the use of laboratory tests to determine the quality of one individual shipment. In this latter case, variations in the laboratory determinations caused by variations in quality between different lots of coal are theoretically eliminated, and the deviations from the average of repeated resamplings of that one lot should be somewhat less than what we have here referred to as the characteristic error for the coal. The fact remains, however, that characteristics of

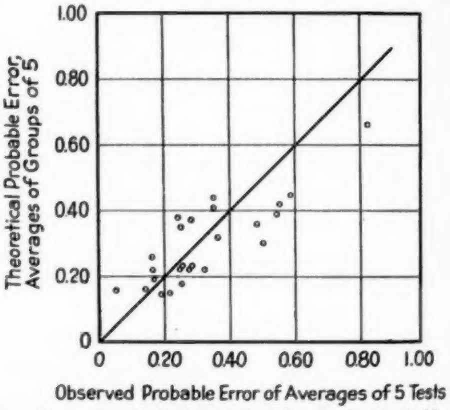

FIG. 5.—Relation of Observed Probable Error to Theoretical Probable Error for Averages of Groups of Five Tests.

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[20] The statistical studies of Grumell and Dunningham and of Morrow and Proctor show similar agreement.

a coal which increase the variations in quality from lot to lot, also tend to increase the probable error which will be found in resampling a given shipment.

It is obvious, therefore, that if an important decision or an engineering calculation of plant performance rests upon a single sample and laboratory test, there is always the chance that the result is among the 1 per cent of tests which approaches the limits of probablity, although more than 80 per cent of a series of tests would fall within twice the probable error, and at least half of them within the probable error. Therefore, when much depends upon a single sample great care should be exercised in the sampling, and even then the chances of error should be recognized.

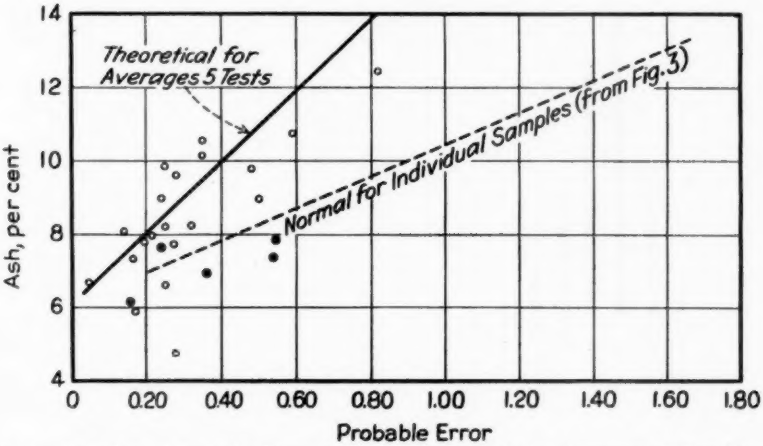

FIG. 6.—Relation of Probable Error to Percentage of Ash for Averages of Five Samples.

Since the larger deviations occur very infrequently, there is a natural tendency to jump to the conclusion that the sampling is at fault and that the particular test can be disregarded as not being "representative" of the quality of the coal. The occurrence of these larger deviations can be predicted, both in magnitude and frequency, from the probable error characteristic of the particular coal. But the occurrence of major deviations, either above or below the average, at greater than normal frequency does bring into question the sampling or testing methods.

Variations among laboratory tests of the same coal, while to some extent due to errors arising out of sampling and testing procedure, are too often and quite indiscriminately assumed to be due entirely to this cause. The fact must not be lost sight of, that coal *is* a variable product. Disregard of this fundamental fact leads to too great reliance upon a single laboratory test, or upon a plant trial of a single shipment and consequently to the unscientific and frequently misleading use of good scientific tools.

Assuming that the probable error found to be characteristic of a given coal cannot be substantially reduced below the norm for ordinary commercial sampling except by elaborate and costly sampling procedure, resort can be had to the use of averages of the tests of a number of samples to secure the desired accuracy. This is particularly useful in comparing the relative quality of two or more coals.

In theory, the probable error varies inversely as the square root of the number of tests averaged. Therefore, if tests of five samples of a coal which has a probable error for individual samples of 0.50 per cent are averaged, the group average may be expected to have a probable error of $\frac{0.50}{\sqrt{5}}$, or 0.22. For the same coal a group average of 10 tests may be expected to have a probable error of 0.16. In practice, the theoretical gain in accuracy is not quite obtained in more than half the cases. This is illustrated in Fig. 5, which shows the relation between the observed probable error for averages of groups of 5 tests for the 25 bituminous coals used for Fig. 3, and that which would be theoretically expected for each coal.

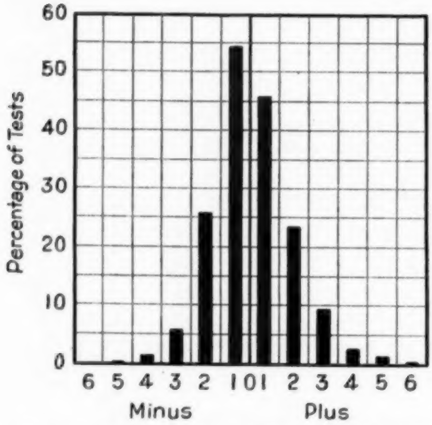

FIG. 7.—Deviation from the Average Percentage of Ash Within Limits of Multiples of Probable Error—Plus and Minus.

640 samples of 25 bituminous coals.

The gain in accuracy is, nevertheless, substantial, as can be seen by comparing Fig. 3 with Fig. 6, which shows the probable error of averages of groups of 5 tests for each of the 25 bituminous coals plotted against the percentage of ash. The curve for individual samples is repeated from Fig. 3 for reference, and the curve passing through the points represents the theoretical probable error calculated from it.

If a coal has a probable error below 0.60, a satisfactory degree of accuracy can be obtained by averaging 5 tests; if the probable error lies between 0.60 and 0.90, an average of at least 10 tests should be used; when the probable error exceeds 0.90, an average of at least 20 samples is needed. For bituminous coal of small size, the following will serve as a rough guide, when the probable error for a coal is not known:

For coals under 8 per cent ash............	average of not less than 5 samples
For coals between 8 and 10 per cent.....	average of not less than 10 samples
For coals over 10 per cent ash............	average of not less than 20 samples

While the distribution of errors within specified limits above or below the average follows the theoretical distribution and can be predicted for a series of samples of the same coal, the distribution is not symmetrical. The number of samples on the minus (low ash) side is always more than half of the whole number of tests, the larger individual deviations appear more frequently on the plus (high ash) side, and usually, but not always, the maximum deviation is found on that side.

If the plus and minus deviations for the 640 tests are separated, this unbalance can be seen readily.

DISTRIBUTION OF DEVIATIONS ABOVE AND BELOW THE AVERAGE.

	PERCENTAGE OF TESTS		
	MINUS, PER CENT	PLUS, PER CENT	PLUS OR MINUS, PER CENT
Within limits of probable error..........	28.7	22.2	50.9
2r....................	48.6	36.2	84.8
3r....................	53.2	43.1	96.3
4r....................	54.2	44.4	98.6
5r....................	54.4	45.4	99.8
6r....................	54.4	45.6	100.0

This distribution is shown graphically in Fig. 7.

The principal reason for this unbalanced distribution, I think, is to be found in the nature of coal mining. Mining methods are designed, with differing degrees of success, to exclude free impurities. Maintenance of any desired quality of product requires constant vigilance and care. Deviation from standard methods of mining or cleaning is more likely to be on the side of some relaxation from the standard, and this results in the more frequent high-ash deviations. If we eliminate the factor of conscious selection in sampling, that is, the conscious inclusion or rejection of visible impurities, which is difficult in sampling small coal (the smaller the coal, the more difficult it becomes), it does not seem that the chance of including an excessive proportion of free impurities in a sample is substantially greater than the chance of excluding them. Sampling errors probably constitute a minor cause of the unbalanced distribution.

The fact that a larger number of tests is normally found on the low-ash side of the average leads to a slightly misleading estimate of the average, which is obtained from a casual inspection of a list of analyses. This needs to be taken into consideration in the interpretation of laboratory tests.

DISCUSSION

Mr. E. S. Grumell[1] *(presented in written form)*.—On my return from a visit to the United States in 1931, I reported to the Directors of Imperial Chemical Industries Limited the lines on which G. B. Gould approached the subject of linking up laboratory tests with the practical utilization of coal. Since then a very considerable amount of research has been initiated in several countries.

Mr. Dunningham and I have studied Mr. Gould's present paper with great interest, and we should like to make the following comments.

The conception of a natural variability in the constituents of industrial coal seems now to be generally accepted. The tentative suggestion that the variability as measured by the probable error would be proportional to the ash content is confirmed by the data presented in Figs. 3 and 4 of the paper. Further, there is, on the whole, reasonable agreement as to the degree of proportionality. Taking the angle between the line through the points and the horizontal as representing the relationship, the angle of Morrow and Proctor's results in Fig. 4 is similar to that for British coals; the angle formed by those of Gould's results in Fig. 3 is somewhat less, although I think the connection of the points might be slightly modified to bring them nearer the same angle. Recent investigations by L. A. Bushell of the Fuel Research Institute of South Africa[2] give an angle almost exactly the same as that of Morrow and Proctor. It seems, therefore, that a relationship between probable error and ash content has now been definitely established.

With regard to the magnitude of the probable error, American coals appear to have lower probable errors than British coals, as is shown in the following table taken from Figs. 3 and 4:

Ash, per cent	Fuel Engineering Company[a]	Morrow and Proctor	British
6	0.25	0.2	0.75
8	0.55	0.45	1.00
10	0.90	0.75	1.20
12	1.35	1.05	1.45

[a] Slightly modified.

I think it desirable that steps should be taken to find the cause of these differences. There are at least two possibilities.

Mr. Gould suggests that the greater variability of British coals may be accounted for by a higher proportion of free impurities. Some time ago T. W. Guy in America emphasized the importance of distinguishing between inherent and free ash. That such a distinction should be made is clearly shown by the work of Bushell. He showed that the average error of South African coals was of the same order as the anthracite-barley-cleaned on Fig. 3 and, therefore, much lower than that of British coals. Now, defining the inherent ash as the ash content of that part of the coal which

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[1] Fuel Economy Section, Imperial Chemical Industries Ltd., Winnington, Northwick, England.

[2] L. A. Bushell, "The Sampling of Coal," *Journal*, Chemical, Metallurgical and Mining Soc. of South Africa, February, 1937.

	MAY 1927–JANUARY 1929	JANUARY 1929–DECEMBER 1930	JANUARY 1931–DECEMBER 1931	JANUARY 1932–DECEMBER 1932
Average ash content	6.58 per cent	7.28 per cent	7.60 per cent	7.11 per cent
Average error	0.44 per cent	1.44 per cent	0.90 per cent	0.85 per cent
Standard deviation (σ)	0.55	1.80	1.13	1.07

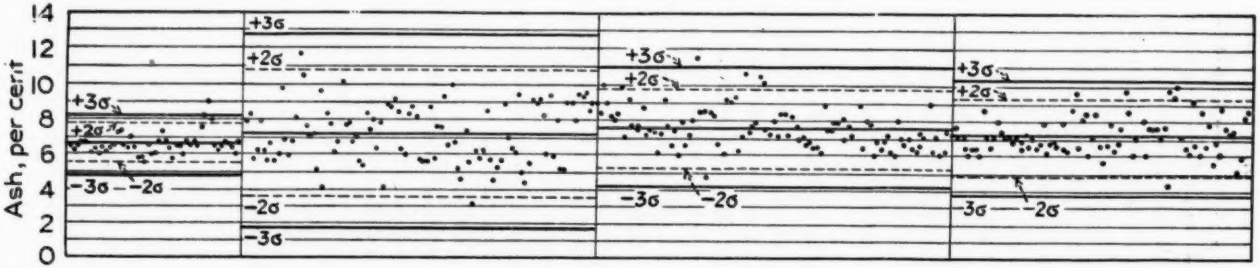

FIG. 1.—Ash Content of Washed Beans.

	DECEMBER 1926–JUNE 1928	JUNE 1928–APRIL 1930	APRIL 1930–DECEMBER 1932
Average ash content	8.97 per cent	8.47 per cent	8.88 per cent
Average error	1.33 per cent	1.27 per cent	0.834 per cent
Standard deviation (σ)	1.67	1.59	1.045

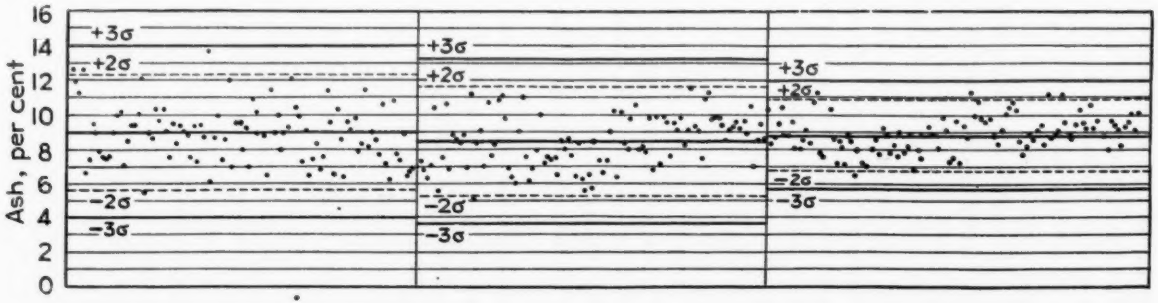

FIG. 2.—Ash Content of Dry Slack.

floats in specific gravity of 1.60, it was found that the South African coals in question contained 8 to 9 per cent of inherent ash, whereas the majority of British coals contain from 4 to 4½ per cent. This undoubtedly accounts for the lower probable error of South African coals. It would be very interesting if similar values could be given for the American coals which have been investigated.

Secondly, size is of some importance. There may be a difference—for the same ash content—of as much as 0.85 in the average error between a rough slack, all passing a 2-in. screen and a smaller, more uniformly graded fuel.

I think that in the days to come it will be necessary, when discussing average error of fuels, to give precise information about size and free ash content.

There is, in addition, another factor of considerable importance which has provisionally been termed "level of control."

Reference has been made to the "characteristic" probable error of a coal. It is probable that such a thing exists, but there is now some evidence to show that it can be modified either by methods of mining or by the degree of supervision exercised in a cleaning plant.

The following example relates to deliveries of a closely graded fuel, cleaned in a wet washery (see the accompanying Fig. 1). Each point represents the ash content of one car. For each period the *average* ash content is much the same. During the first period the washery was under strict supervision; the variations from wagon to wagon were very small and the average error only 0.44. During the next period (after a change of management) the ash content of individual cars varied considerably and the average error jumped to 1.44, subsequently improving to 0.85. It is clear that the "level of control" in the washery changed and influenced the variability of the product without, however, appreciably affecting the *average* ash content.

Something similar, but possibly smaller in degree, may occur with coal drawn direct from the mine, depending, presumably, upon the method of mining. This is shown in the accompanying Fig. 2 for a 1-in. to nothing dry uncleaned slack.

It is, therefore, possible for coal from the same seam but from different collieries to have different average errors according to the method of mining the coal.

MR. A. C. FIELDNER.[3]—Sampling and the interpretation of sampling is of great importance. Messrs. Grumell and Dunningham, of England, took up this question in recent years. They reexamined the work of E. G. Bailey originally conducted in this country in 1909 and applied it to British coals. Mr. Gould has given a critical analysis of recent work of American investigators in this field, and we hope that we shall get more information that we can use in application to our own coals, so that producers and consumers will take into consideration the importance of taking a number of samples and averaging them rather than depending upon a single sample of coal.

MR. O. O. MALLEIS.[4]—I shall comment on only two points in Mr. Gould's paper.

I was rather disappointed that no attempt was made to point out possibilities for practical use of the calorific value of the "pure" coal. This term, at the present time, has not gotten very far out of the laboratory. The fuel chemist, of course, has long made use of the dry, ash-free B.t.u. value as a check of the

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[3] Chief, Technologic Branch, U. S. Bureau of Mines, Washington, D. C.

[4] Manager, Inspection Division, Appalachian Coals, Inc., Cincinnati, Ohio.

accuracy of his calorimetric determination. From both the purchase and utilization standpoints, the establishment of reliable average dry, ash-free B.t.u. values for the various seams of bituminous coal should be of considerable practical value even if it would be necessary to have more than one value for a very extensive seam of coal. In the southern high-volatile coal field, the range in dry, ash-free calorific values is from approximately 14,750 to 15,500, or a range of about five per cent, due to differences in the B.t.u. value of the "pure" coal. Certain useful performance characteristics undoubtedly could be associated with various B.t.u. values of the "pure" coal which should be of value for evaluating coals from both the combustion and carbonization standpoints.

The other point is with regard to tolerances for test values. In order to be more certain of keeping within the allowable tolerances for the usual coal determinations, it is my feeling that fine grinding (even to as small as 4 mesh) before the first reduction in quantities is the procedure to follow not only when comparing results for several gross samples for one shipment but also for single samples from a series of shipments of one grade of coal from the same mine. Possibly finer grinding of the entire gross sample will result in smaller gross samples without any increase of present tolerances.

MR. A. W. GAUGER.[5]—Referring to the sampling procedure in power plants, many such plants, particularly the smaller ones, are not equipped to take samples in accordance with the procedures established by the U. S. Bureau of Mines and the National Bureau of Standards. In fact many operators in small power plants have not the slightest idea as to what these procedures are. A campaign of education is necessary, and discussion of this sort should be disseminated broadly.

So far as the reduction of the sample for the final analysis is concerned, it was unbelievable to me that any chemist who was making proximate analyses would not know the importance of a careful reduction of the sample. I ran into considerable difficulty due to very erratic results of the percentage of ash and on such calculations as the so-called H-value. When traced down, the difficulty was due to the fact that in the reduction of the sample to 60 mesh, everything that went through the sieve came up to the laboratory for analysis but there was a residue that was discarded. We must understand that the components of the coal mixture have varying resistance to grinding. Hence, it is very important in the reduction of the sample for analysis that it all be reduced to 60 mesh and that it all go into the bottle that goes to the laboratory.

MR. A. W. THORSON.[6]—The paper by Mr. Gould, showing statistical data on coal analyses, conveys to the writer that a number of samples and their analyses must be available before any conclusion can be formed as to coal properties. Too often, judgment of coal properties is based on one analysis of one sample which may have been improperly taken. I should like to place emphasis on Mr. Gauger's remark that an intensive educational campaign should reach the people who do not seem to appreciate the necessity for very careful sampling and analyzing.

The A.S.T.M. standards have been set up and are being constantly improved by the efforts of the various committees. A great deal of information is available

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[5] Professor of Fuel Technology, and Director of Mineral Industries Research, The Pennsylvania State College State College, Pa.

[6] The Detroit Edison Co., Detroit, Mich.

to the users of coal. The problem is apparently one of disseminating this knowledge and encouraging its use.

In our own plants, we went to mechanical sampling several years ago. We are now bringing our procedure in line with the tentative revision of the Standard Method of Sampling Coal (D 21 - 16) proposed by Committee D-5 on Coal and Coke, and accepted by the Society at this meeting[7]—the first guide that has been available to users of mechanical methods.

MR. C. A. REED.[8]—Mr. Gauger referred to the large number of people who use coal as against the large tonnage users. I realize that the large consumer probably does not need much education or much help because he has worked out his own problems, but, as Secretary of the Bituminous Coal Research, Inc., I find that among 1500 coal companies, many of the salesmen, managers, engineers, or others who write to us, are writing to a large extent about customers who use less than 10,000 tons of coal a year, inquiring as to how they can help those customers stop doing something wrong and start doing something right. While I was with the Pittsburgh Coal Co., we in the field found that we had to educate power house engineers, efficiency engineers and coal handlers in the method of taking a sample. I recall one instance where a company that used 500,000 tons of coal a year did its sampling in this manner—and this was only a very few years ago. A man with a bag strapped to his back would step up to the side of a railroad car and with a shovel scoop up a shovelful of coal from the corner of the car and dump it into his bag; he would repeat that with ten cars. That represented a composite sample, and there would be a wide variation of analysis even from a mechanically clean coal. In talking quite seriously to the purchasing agent, who was the only man you could talk to, his answer was: "It costs too much to take the type of sample you suggest," and my answer was, "Then why take the samples at all?" But I want to come back to the point that if we can get the information broadcast to the man who uses less than 10,000 or less than 20,000 tons of coal—that he should either take the samples properly or not at all. In lieu of sampling he should see what his steam cost is. We knew, for example, in certain of our mines, that we had a control of within one-half of 1 per cent of ash on, we will say, minus 2-in. coal. Yet we had customers who were getting a car sample of as much as 2 or 3 per cent variation on probably a bushel basketful out of 50 tons. Some of those customers, who were not necessarily large consumers, had in their plant an unlimited number of gages and other measuring devices for testing and sampling equipment which they sold—tensile strength machinery and speed machinery and efficiency machinery to test the equipment that they were putting out. To overcome the difficulty of having people say, "Your coal is no good," we probably spend hundreds of dollars on individual small consumers whereas the large consumer could easily be shown. If, however, your steam cost does not check with the analysis you are getting you must know your coal sampling is not correct.

MR. G. B. GOULD.[9]—Mr. Malleis suggests that more use should be made of the moisture- and ash-free B.t.u. value. This is a very useful figure for the fuel technologist, but I fear that any attempt to popularize its use would only lead to

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[7] *Proceedings*, Am. Soc. Testing Mats., Vol. 37, Part I (1937); also 1937 Book of A.S.T.M. Tentative Standards, p. 1583.

[8] Director of Engineering, National Coal Assn., Washington, D. C.

[9] President, Fuel Engineering Company of New York, New York City.

confusion in the minds of both sellers and buyers of coal, many of whom still have difficulty understanding the more directly usable terms: "B.t.u. as received" and "B.t.u. dry."

We are indebted to Mr. Grumell for his discussion, for it was his work which has given us an entirely new light on the variability of coal and started such a valuable new line of thought on coal sampling methods. He refers to the recent work of Bushell, which I did not have an opportunity to study before this paper was written. Bushell presents some valuable experimental data on the relation between sample accuracy and the size of increments, and also has devised an ingenious mathematical method of expressing the variability of coal, based upon the percentage of free impurities (sink at 1.6), the percentage of ash in them, and the percentage of float at 1.3. His formula can be applied to our coals, though possibly the constant which is suitable for South African coals may need to be modified for our use. Mr. Grumell presents some interesting data supporting the view that the "characteristic" error for a coal is somewhat dependent upon the methods of mining and preparation. He calls attention to the fact that the magnitude of the error in the South African coals agrees quite closely with the magnitude of the errors for our No. 3 buckwheat anthracite. We did not determine the inherent ash in the anthracites that are represented by the points on Fig. 3, but similar coals from other data we have in the office indicate that the inherent ash in our mechanically cleaned anthracite very closely agrees with the inherent ash in the South African coals.

This discussion has taken a slightly different turn from the line of thought that I had in this paper, because I purposely avoided sampling methods, which I think need to be revised, but this sampling discussion, which is something different from the variations in the coal itself, brings up some points which I think ought to be a little better clarified. As may be seen from the curves, occasional extreme variations are found. The agreement of our studies of thousands of tests in many series with Morrow's and Proctor's data indicates that if any extreme variations are taking place in sampling, they are very few in number. It is very easy for one individual sample that deviates from the average by 3 or 4 or 5 times the probable error for that coal, to attract a great deal of attention and create the impression that all sampling is very unreliable. Sampling can be greatly improved, but it is not anywhere nearly as bad as some people think. That may be seen statistically by taking the data of Fig. 3, because there we have the variations from one lot to another lot of the same coal with different sampling, and sampled by people in many cases who were quite inexperienced; even so the tolerances are only what we show on that chart. When you look at thousands of tests and study them you get a very different view of this thing, and come to realize that sampling generally is not so bad as it looks—but I agree that once in a while it looks pretty bad. In that connection, recently we made an experiment which, as a matter of fact, is not complete yet, on the resampling of one lot of 500 tons of coal. It was a 9 per cent ash, $1\frac{1}{4}$ in. Eastern bituminous nut and slack. According to our normal curve in Fig. 3 a coal of that kind would show a variation from lot to lot, a normal probable error of around 0.70 to 0.75. We sampled that same pile of coal twenty times, individual gross samples. They were all 40-lb. gross samples, divided once by a rifle without any crushing at all, and from that point down (from 20 lb. down) they, of course, were properly reduced in size by grinding in the laboratory. Those 20 samples showed a probable error of 0.15, which indicates that sampling can be done and within reasonable tolerances without impossible methods of sampling. We need more investigation of that point for our American coals. That is just one example to show that good sampling is not a very difficult thing.

Mr. Reed refers to some specific cases of bad sampling, and makes a plea for more widespread information on sound sampling methods, especially among the smaller consumers. This is needed, but first reasonable tolerances should be determined, and methods of sampling made as simple and easy as those tolerances permit. He makes one point which needs further examination, when he says "In lieu of sampling he (the consumer) should see what his steam cost is." If records of power plant performance in these same small plants are studied as critically as the sampling methods, it will be found that steam cost calculations are subject to serious and frequent errors.

THE SIGNIFICANCE TO THE CONSUMER OF SULFUR IN COAL

By Henry Kreisinger[1]

This paper deals with the significance of sulfur in coal as viewed by the consumer who uses the coal as fuel for making steam. Sulfur is an undesirable element in coal, and the consumer looks with much suspicion on coals high in sulfur. He feels that high-sulfur coal is likely to give him trouble from clinkers on grate, slagging of furnaces, slagging and fouling of heat-absorbing surfaces, corrosion, and pollution of atmosphere by obnoxious gases. High-sulfur coal is also apt to give trouble from spontaneous combustion in coal storage pile and coal bunker. Even when the price of high-sulfur coal is substantially lower than that of low-sulfur coal, the experienced consumer will not make contracts for high-sulfur coal without first giving the coal a thorough trial to convince himself that the difficulties encountered in using such coal will not more than offset the advantage of the lower price.

Sulfur in coals occurs mainly as sulfide of iron, commonly known as pyrites, and as organic sulfur. A small amount of it also occurs as sulfates. Some of the troubles mentioned are caused directly by the sulfur; others are due to the iron with which a large part of the sulfur is combined.

In a boiler furnace the sulfide of iron generally burns to iron oxide and SO_2 or SO_3. In a reducing or a partly reducing atmosphere all of the iron and sulfur may not be completely oxidized and some ferrous sulfide, low oxides of iron or even metallic iron may be formed. The organic sulfur also burns to SO_2 or SO_3. The sulfates decompose at the high furnace temperature into metallic oxides and SO_2 or SO_3. The SO_2 and SO_3 are gases and pass with the gaseous products of combustion to the stack. The metal oxides and the partly burned sulfur compounds become part of the ash and are removed with it.

Clinkering and Slagging:

The formation of clinker in the fuel bed of hand- and stoker-fired furnaces, slagging of furnace walls, and slagging and fouling of heat-absorbing surfaces in steam-generating units are the most serious troubles the coal consumer fears with high-sulfur coal. Such troubles cause much additional work in the boiler plant, and increase the maintenance of the equipment; they also seriously reduce steaming capacity of the plant.

The sulfur and the iron, with which the sulfur is combined are apt to lower the softening temperature of the ash and thereby cause clinker in the fuel bed, slagging of the furnaces, and slagging and fouling of heating surfaces. Figure 1 is intended to show to what extent the sulfur content in Pennsylvania coals affects the softening temperature of ash. The chart has been prepared from *Bulletin 209* of the U. S. Bureau of Mines.[2] It shows that coals with ash

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[1] Engineer in Charge of Research and Development, Combustion Engineering Co., New York City.

[2] W. A. Selvig and A. C. Fieldner, "Fusibility of Ash from Coals of the United States," *Bulletin 209*, U. S. Bureau of Mines (1922).

of high softening temperature generally have low sulfur content, but not all low-sulfur coals have high fusion temperature ash. However, most high-sulfur coals have low softening temperature ash. Although no definite relation between sulfur content in coal and softening temperature of ash can be deduced, the chart justifies the suspicion of the coal consumer that high-sulfur coal is likely to cause clinker and slag troubles. The consumer is opposed to contracting for high-sulfur coals without first giving the coal a thorough trial to find if such coal can be used economically in his boiler plant.

When the coal is burned on grates either in hand- or stoker-fired furnaces, the clinker troubles are usually less serious if pyrites occur in large lumps or disks than when they are uniformly distributed through the coal. The large lumps are mostly separated from coal, and, because they are much heavier than the coal, during the process of combustion they sink to the grate out of the zone of high temperature, and may not reach the softening point. There is also less sulfur and iron carried by the gases into the boiler and, therefore, there is less likelihood of slagging the heating surfaces of the boiler. This is probably the reason why some high-sulfur coals may give satisfactory results as far as clinker and slagging of boiler is concerned. When such coals are burned in pulverized form most of the pyrites as well as the coal are pulverized and are then uniformly distributed through the coal and may cause slagging of the furnaces and of the boiler.

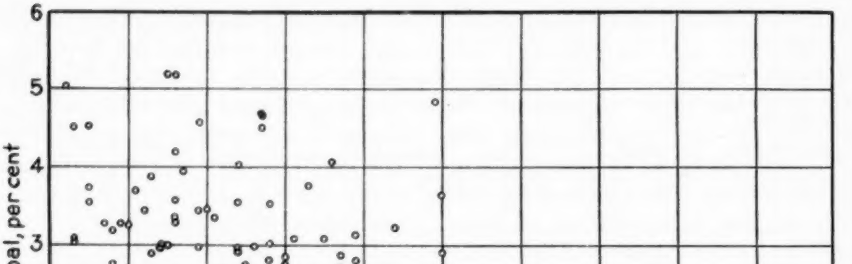

Fig. 1.—Relation Between Percentage of Sulfur in Coal and Softening Temperature of Ash. Pennsylvania coals.

Clinkering in the fuel bed, and slagging of furnaces and of the heating surfaces increases the work of operating the boilers. The clinkers and slag must be often removed from the furnaces by hand tools. Slagging and fouling of the heating surfaces requires frequent operation of soot blowers and the use of hand lances. The capacity of the steam-generating equipment under such operation is usually reduced because the clinker and slag and their removal hinder the combustion of fuel. The clinker and slag damage the grate and the furnace. Thus, the maintenance of the equipment is increased. Among the first questions that high-sulfur coal brings to the consumer's mind are those of increased operating and maintenance cost, and of reduced capacity of steam-generating equipment.

Corrosion:

The products of oxidation or decomposition of the sulfur compounds in coal are corrosive, and may cause rapid deterioration of all metallic parts with which these products come in contact. The corrosion may start in the coal-handling equipment and coal bunker where the oxidation of sulfur in presence of moisture and air produces sulfuric acid which attacks iron or concrete. The sulfur which remains in the clinker and ash eventually reaches the ash hopper where the refuse is usually quenched. The water used in quenching picks up some of the sulfur compounds from the refuse and forms dilute acids which corrode the iron parts of the ash hopper and ash-handling apparatus. The gaseous products of combustion and decomposition of the sulfur are carried through the boiler setting with other gaseous products of combustion. When their temperature is reduced in the presence of water vapor they form acids which corrode and foul the economizer, the air heater, flue ducts, and steel stacks. Water vapor is produced by moisture and the hydrogen in coal, the quenching of refuse in the ash hopper, and the operation of soot blowers to remove the fouling from the surfaces. The removal of fouling with compressed air reduces the amount of water vapor in gases and thereby the acid formation, but the use of compressed air requires additional equipment for compressed air and power for its operation. Generally the higher the proportion of water vapor in the products of combustion the more serious is the corrosion trouble.

Low temperature of the products of combustion that favors the condensation of water vapor and the formation of acids is obtained when the boiler started or shut down, and when it banked. It may also be produced by feeding the economizer with water at too low temperature. Low temperature generally occurs in air heaters at the point where the air enters the heater. Condensation of water vapor in presence of SO_3 starts at higher temperature than the dew point corresponding to the partial pressure of the water vapor.

In this connection it may be stated that fouling and corrosion of economizer and air heater are usually more serious with high-sulfur fuel oil than they are with high-sulfur coal. The explanation probably is that fuel oil contains a greater percentage of hydrogen and more water vapor is formed by the combustion of oil. The sulfur in oil occurs mostly as organic sulfur and the greater part of it may burn to SO_3.

Air Pollution:

The products of combustion and decomposition of the sulfur compounds flowing out of the stack give the waste gases obnoxious odor thereby causing objectionable pollution of the air. Breathing such polluted air is very unpleasant and irritating even to healthy people, and if the plant is located near or in a town, there may be serious complaints against such air pollution. The sulfur gases are destructive to stone and concrete buildings and to paint; they are also destructive to plant life. In the London district in England, power plants are required by law to remove the sulfur compounds from stack gases before discharging them into the atmosphere. In the United States, the subject of air pollution by sulfur gases is kept alive by discussions at all smoke-abatement and pure-air meetings. While no laws have so far been proposed for compulsory removal of sulfur compounds from stack gases, much work has been done to develop an inexpensive method for such removal. The problem of air pollution by sulfur gases may be of considerable importance requiring serious consideration by the coal consumer whose plant is located where it might cause complaints.

Storage of Coal:

High-sulfur coal does not store well. Slow oxidation of the sulfur lowers the heat value of the coal and increases the danger of spontaneous combustion. Even if the coal is stored in large coal bunkers for immediate use, high-sulfur coal may cause troublesome fires before the coal is used up. If a coal consumer has to store part of his coal requirements in a pile to guard against coal shortage, he is very likely to avoid high-sulfur coal.

DISCUSSION

Mr. P. Nicholls.[1]—Mr. Kreisinger, in his presentation repeatedly implied that sulfur produces clinkering; although I know that he uses this expression as a contraction for "the iron combined with sulfur as pyrites," yet it may have misled some. The only form in which sulfur is found in slags is as entrained particles of ferrous or calcium sulfide (FeS or CaS) which have not had an opportunity to oxidize. We have many published and unpublished analyses of slags of all types; none has shown high sulfur and usually the content is 0.1 per cent or less, which is below 1 per cent of the sulfur in the coal. The highest has been from slags from fuel beds which have been less fused, but even then the maximum was 3 per cent of the sulfur in the coal.

I would grant that there would be worse general clinkering if the pyrites are uniformly distributed through the bed, but I have seen more trouble due to its being concentrated in lumps. One had to accept any type of coal during the World War; at a certain plant the coal received contained lumps of pyrites with the result that they would melt, run through the grates, and I have seen stalactites 6 in. or more in diameter at the top and 2 ft. long hanging from the grates, so that the furnaces had to be shut down and the grate bars chipped out.

Another type of trouble because of sulfur has occurred in slag-tap furnaces: Particles of ferrous sulfide reach the bed of molten slag without being oxidized; being denser than the slag, they sink and form a molten mass. When the slag is tapped the molten sulfide may be exposed to the air and it will burn, causing very objectionable and, in some instances, dangerous conditions.

There has also been considerable trouble because of the formation of masses of metallic iron at the bottom of the beds of slag-tap furnaces. My deduction has been that this is directly due to the ferrous sulfide. Particles reach the surface of the bed together with particles of carbon and are reduced to iron; these particles, being denser than the sulfide, sink too low in the bed to be removed when tapping and thus gradually accumulate and ultimately have to be drilled out.

For some purposes, a knowledge of the quantity of sulfur in the coal is not sufficient and the forms of sulfur should also be known.

Mr. R. L. Rowan.[2]—I agree with most of the things that Mr. Kreisinger attributed to this, shall we say, Public Goat No. 1. All of those things undoubtedly do take place in actual practice. I do not know that I can agree with him entirely in laying all spontaneous combustion in storage piles of coal to sulfur. I think there are many other factors that enter into it, as he undoubtedly will agree. There has been too great a tendency in many cases, whenever we have had any clinker trouble or heating in a storage pile, to say, "Oh, well, the coal has too much sulfur in it."

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[2] Fuel Engineer, General Coal Co., Philadelphia, Pa.

When we analyze coal we report the total sulfur. While for metallurgical purposes it is possibly correct that that is indicative of what might be expected, certainly as far as clinkers are concerned I think if we also reported the pyritic sulfur content we would have a closer index of what might take place in the fuel bed, especially in underfeed stokers and hand-fired furnaces. Probably that would not be so with powdered fuel, because you would get some slagging and corrosion with all of your sulfur, but I think it is unfortunate with any coals where the pyritic sulfur content is not more than half the total sulfur content, it is reported as a coal of a certain sulfur content, yet only about half of it will cause any trouble. I believe it would help in our practice if we could have more published data in that respect.

MR. R. A. SHERMAN.[3]—One item has occurred to me that Mr. Kreisinger did not discuss in relation to difficulties from sulfur in coal—it is the annoying concentration of SO_2 in the boiler rooms, particularly on the levels above the stoker hoppers. One never wants to have gases backing up from the stoker hopper, but it very frequently occurs and even a small concentration of SO_2 can be very distressing to men who have to work in it. Similarly, a high sulfur coal will frequently leave enough sulfur in the ash and clinker to be very unpleasant to the men working on dumping of ash hoppers.

MR. G. C. SQUIER.[4]—At what temperature in flue gases will SO_2 or SO_3 unite with H_2O to form either sulfurous (H_2SO_3), or sulfuric (H_2SO_4) acid?

MR. HENRY KREISINGER[5] (*author's closure*).—Mr. Nicholls' comment is made from the standpoint of a scientist who analyzes a problem in detail in order to ascertain the true causes of the difficulties which the problem presents. He is right from his point of view. My paper was written from the point of view of a coal consumer, who generally makes his preliminary coal selections from proximate analyses, heat value, sulfur content, and in some cases the fusibility of ash and the grindability of coal. The composition of ash and the form in which sulfur occurs in coal is not usually given. He compares the heat value first, and then the ash, volatile matter and sulfur content. High sulfur content in coal always raises a suspicion in his mind that the coal will give him trouble. The coal remains under suspicion until it is tested under actual operating conditions and proves itself to be reasonably good fuel. Mr. Nicholls thinks of the troubles sulfur actually gives under various conditions, whereas my statements refer to the troubles that the average consumer thinks sulfur may cause. In other words, to the coal consumer sulfur in coal is an indicator of probable troubles.

Mr. Rowan agrees in general with the statements in my paper; he differs as to the degree to which sulfur is responsible for the troubles enumerated in the paper. While in some cases sulfur may not be the principal cause of the troubles, nevertheless it is a contributing factor. One thing is certain, it contributes nothing to make the coal a better fuel. Even if we had a complete analysis of the ash and the exact composition of the compounds in which sulfur occurs, we could not be sure with our present knowledge of the subject that high-sulfur coal would make a good fuel. We would want a practical demonstration by a good trial under actual operating conditions.

Mr. Sherman brought an additional factor against sulfur in coal and that is

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[3] Fuel Engineer, Battelle Memorial Institute, Columbus, Ohio.

[4] Assistant to President, J. F. Massey and Co., Inc., New York City.

[5] Engineer in Charge of Research and Development, Combustion Engineering Co., New York City.

the annoyance of SO_2 to boiler plant operators in case of leakage of gases from boiler settings, and in the handling of the ash pit refuse. Such annoying experiences necessarily prejudice the boiler room operators against high-sulfur coal, and they will be reluctant to do their best to obtain good results with such coal.

Mr. Squire asked about the formation of SO_3 and its combination with H_2O to form sulfuric acid. There is no exact data on that subject, but from what information we have we can say that about one third of the sulfur burns to SO_3. Condensation of water vapor in presence of SO_3 may occur even at 300 F. which is far above the ordinary dew point; the higher the concentration of SO_3 and water vapor in the gases, the higher will be the temperature at which water vapor condenses and absorbs SO_3 to form sulfuric acid.

More sulfuric acid seems to be formed with high-sulfur fuel oil than with coal, because of the higher concentration of water vapor in the products of combustion.

THE SIGNIFICANCE OF ASH SOFTENING TEMPERATURE AND ASH COMPOSITION IN THE UTILIZATION OF COAL

By A. W. Gauger[1]

Introduction

The subject of the practical significance of the softening temperature of coal ash as measured by the standard A.S.T.M. test method[2] is one that should be approached in a spirit of humility and caution. A long series of illustrious technologists has attacked this problem with two general objectives in view. The first of these, the attainment of a standard method of measurement, has been accomplished largely through the efforts of A. C. Fieldner and his coworkers in the U. S. Bureau of Mines. The second of these, the relating of the results obtained by the standard method to the very practical problem of predicting the probable severity of clinker formation to be anticipated in the case of any given coal, equipment, and set of firing conditions, is still far from conclusive solution.

At present, it is generally accepted by fuel technologists and engineers that there is no simple, direct correlation between ash softening temperature and clinker trouble. Thus, Nicholls and Selvig[3] state that:

> No one simple measure of the nature of the ash, such as its fusibility determined by an arbitrary method, can be expected to predict closely relative values of troubles resulting from a complex ash passing through a set of conditions in which the temperature, time of exposure to that temperature, and travel of the ash are undefined, uncertain, and dependent on factors that are also variable.

More recently, Kreisinger[4] has written:

> Ash fusibility test gives an indication of a probable clinker and slag trouble, although it is not a positive criterion.

This conclusion is not surprising in view of the fact that the temperatures in the fuel bed of a modern power plant are hot enough to soften the ash of almost any coal. On this subject Ricketts[5] has the following to say:

> The writer's observations indicate that, regardless of the fusion temperature of the ash, all of the ash particles not closely associated with a considerable percentage of unburned carbon, whether they are lifted from the fuel bed by the draft or discharged into the ash pit, have been fused. Logically, they should be because, in general, the temperature of combustion in the fuel bed is 2800 to 2900 F.; most coal ashes will soften under such temperature conditions.

This paper is concerned with the relation of ash softening temperatures and

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[1] Professor of Fuel Technology, and Director of Mineral Industries Research, The Pennsylvania State College, State College, Pa.

[2] Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 – 33), 1936 Book of A.S.T.M. Standards, Part II, p. 407.

[3] P. Nicholls and W. A. Selvig, "Clinker Formation as Related to the Fusibility of Coal Ash," *Bulletin 364*, U. S. Bureau of Mines (1932).

[4] Henry Kreisinger, "Value of Coals as Steaming Fuel as Indicated by Their Chemical and Physical Properties," *Contribution No. 85*, Class F, Coal Division, Am. Inst. Mining and Metallurgical Engrs. (1935).

[5] P. Nicholls and W. A. Selvig, "Clinker Formation as Related to the Fusibility of Coal Ash," *Bulletin 364*, U. S. Bureau of Mines, p. 69 (1932).

the composition of coal ash to practical applications of coal. The author appreciates the many-sided aspects of the subject, and hopes that the discussion during this symposium will constitute a searching examination of the entire problem. In the preparation of the manuscript, previously published work on this subject has been freely utilized; in addition to this, the author also makes grateful acknowledgment to a large number of technologists and engineers who have generously cooperated by furnishing helpful suggestions, data, and by reading and criticizing the preliminary manuscript.

GENERAL ASPECTS OF CLINKER FORMATION

Since this symposium deals with the practical aspects of laboratory tests, it seems logical to seek first an understanding of the reasons for making the standard softening temperature measurement. These are set forth in the early publication of Fieldner, Hall, and Feild[6] which opens with the sentence:

> As a safeguard against excessive clinker troubles, specifications for the purchase of coal can be drawn to include the "softening" or "fusing" temperature of the ash.

Since the ash softening temperature measurement is intended to be an index to "clinker trouble," it is of importance to have at the outset some sort of an understanding of what is meant by "clinker trouble." A clinker is defined by Webster as:

> Stony matter more or less vitreous or fused together, as that formed from impurities in coal slag.

The term comes from the Dutch, and referred originally to a vitreous mass such as a brick which had been burned so hard as to give off a ringing sound when struck.

Clinker trouble, however, involves more than the mere formation of a clinker. It is conceivable that a power plant might be of such construction or may be so operated as to break up and slough off any sintered or vitreous materials that might result from the ash of the coal. In such an event the management would not have clinker difficulties and would not be interested in *any* standard test of a property designed as an index to such difficulties. In other words the important criterion in this connection is operation of the plant without difficulties and not the mere formation of a sintered or fused mass of ash. We have, therefore, to consider not only the properties of the coal and its associated mineral matter, but also the type of coal-burning equipment and the firing practice.

It is of interest to list a few of the many factors which may be summed up under four general heads:

1. Properties of coal including rank, percentage of ash and moisture, ash softening temperature, composition of ash, caking and coking properties of coal, and size-consist of coal.
2. Types of fuel-burning equipment including hand fired, underfeed and overfeed stoker fired, traveling grate, pulverized coal fired, both dry bottom and slag tap.
3. Firing practice, including rate of burning, cleaning of fires, control and distribution of air, preheating of air, etc.
4. Human factors.

These by no means constitute all the factors that are of importance in clinker formation; furthermore, they can be broken down into simpler classifications almost without end. Nevertheless, those listed are ample to indicate that clinker

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[6] Arno C. Fieldner, Albert E. Hall, and Alexander L. Feild, "The Fusibility of Coal Ash and the Determination of the Softening Temperature," *Bulletin 129*, U. S. Bureau of Mines (1918).

trouble is a cooperative affair and may be ascribed to no single cause which will cover all cases.

REASONS FOR FAILURE OF ASH SOFTENING TEMPERATURE DETERMINATION

Any discussion of the reasons for a lack of correlation between ash softening temperature and clinkering tendency of coal ash must give consideration to at least four factors in connection with the mineral matter in coal and the standard test method. The first of these relates to the precision of the standard test, which is entirely empirical and recognizes a tolerance of considerable magnitude within the same laboratory as well as between different laboratories. Thus the permissible variation on a given sample within the same laboratory is 30 deg. Cent. (54 deg. Fahr.) and between different laboratories 50 deg. Cent (90 deg. Fahr.). With tolerances of this magnitude in the laboratory method it is obvious that correlation between performance and laboratory results cannot be expected to recognize differences in ash softening temperature of less than 180 deg. Fahr.

A second factor concerns the variations that occur in the coal samples themselves. Morrow and Proctor[7] have reported the results of an intensive study of variables in coal sampling which indicate for ash softening temperature that the probable error in the case of car samples, if a sufficient number of increments is taken, will be well within the tolerance of the standard test but the maximum deviation between individual samples may be as high as 500 deg. Fahr. for certain sizes.

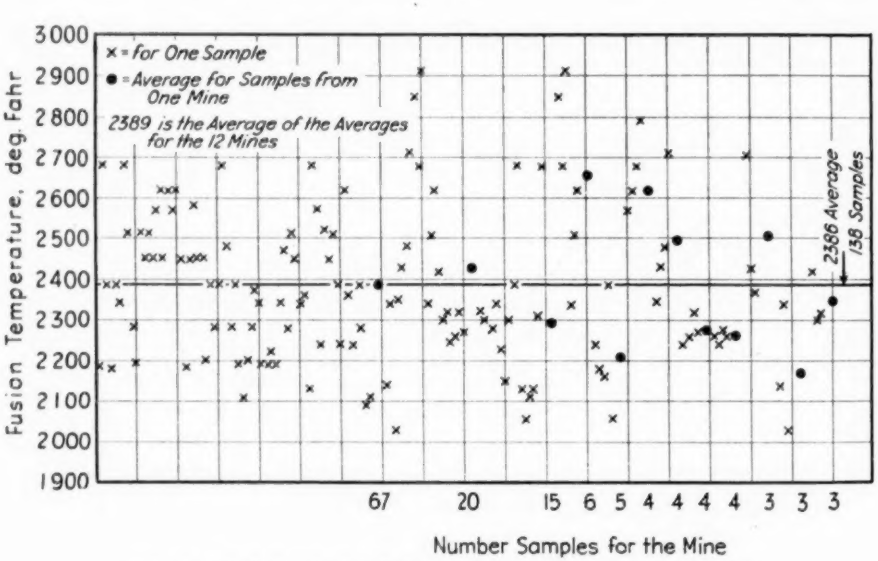

FIG. 1.—Variation in Ash Softening Temperature of Face Samples.

The author is indebted to S. H. Viall for Fig. 1, which shows the variations in the ash softening temperature of face samples from the Indiana Fourth Vein. Viall believes that analyses of at least 20 samples of commercial shipments of a given size are required before one is

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[7] J. B. Morrow and C. P. Proctor, "Variables in Coal Sampling," *Proceedings*, Am. Inst. Mining and Metallurgical Engrs., Vol. 116, pp. 227–269 (1936).

reasonably close to an average value for that coal. While the analysis of the first sample might coincide exactly with the average value, it might also be exceptionally high or exceptionally low. Figure 1 emphasizes the need for numerous determinations before average conditions are known.

A third factor which is closely related to the second is concerned with the variations in the mineral matter in coal. The mineral composition of the coal is related in a complex way to the ash softening temperature. Some knowledge is available on this subject.[8] The mineral composition is also related in a complex way to clinker formation, but unfortunately there are few data on this problem. Experience in these laboratories has shown that there may be a wide variation in the mineral content of samples separated into fractions of different specific gravity by float and sink methods, a fact which is not surprising since the minerals vary widely in specific gravity.

TABLE I.—VARIATION IN ANALYSIS OF HIGH-GRAVITY FRACTIONS OF 0 TO 4-IN. RAW COAL.
Four coals separated into 1.60 to 2.00, 2.00 to 2.60 and 2.60 sink specific gravity fractions.

Specific Gravity	Per cent by Weight	Volatile Material, Moisture- and Ash-Free, per cent									Fusion Temperature, deg. Fahr.
		Ash	Sulfur	Moisture	Air-Dry	Free	SiO_2	Al_2O_3	Fe_2O_3	CaO	
Coal No. 1:											
1.60 to 2.00	16.2	49.7	7.65	0.4	21.9	43.9	49.0	25.0	20.3	1.5	2370
2.00 to 2.60	74.0	77.0	3.40	1.0	12.5	56.8	60.1	28.8	8.6	0.9	2620
2.60 sink	9.8	68.7	23.90	0.4	18.2	58.9	32.0	16.6	44.6	2.7	2395
Coal No. 2:											
1.60 to 2.00	16.9	48.8	5.20	0.5	21.5	42.4	49.0	25.8	15.4	3.9	2320
2.00 to 2.60	76.5	76.1	5.35	0.8	12.8	55.4	57.5	27.7	10.6	0.7	2580
2.60 sink	6.6	57.6	31.35	0.2	22.2	52.7	4.6	6.5	85.6	1.1	2495
Coal No. 3:											
1.60 to 2.00	20.6	50.4	3.65	0.4	19.7	40.0	48.4	23.7	12.3	5.6	2335
2.00 to 2.60	77.5	79.5	1.90	0.5	12.0	60.0	60.1	28.9	5.5	2.0	2625
2.60 sink	1.9	57.7	34.75	0.1	22.0	52.1	10.3	7.5	79.2	1.8	2530
Coal No. 4:											
1.60 to 2.00	19.7	49.9	2.10	0.4	19.9	40.0	49.7	25.1	8.6	7.2	2240
2.00 to 2.60	78.8	79.3	2.30	0.6	11.8	58.7	57.9	28.0	6.5	2.7	2525
2.60 sink	1.5	61.5	34.65	0.2	22.3	58.2	11.9	10.3	73.8	1.9	2495

Petrographic study of ten samples of coals from central and western Pennsylvania and from the Pocahontas seam of West Virginia[9] revealed the presence of pyrite, kaolin, chlorites, muscovite, quartz, calcite and gypsum in all of them. Similar studies have been conducted by Ball and others[10] in the Illinois State Geologic Survey. Two columns were studied from the Herrin No. 6 coal bed in Franklin and Washington Counties, Illinois, and three blocks each from the Upper Freeport bed, Allegheny County and Pittsburgh bed, Allegheny and Fayette Counties. Thiessen, Ball and Grotts conclude from these studies that the separable mineral matter from Illinois and western Pennsylvania coals is composed of over 95 per cent detrital clay, kaolinite, calcite, and pyrite, with other minerals unimportant.

Such a sweeping conclusion is unwarranted on the basis of a study of six Pennsylvania coals and two Illinois coals, for experience has shown that the relative proportions of the various ash-

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[8] Thomas G. Estep, Harry Seltz, Henry L. Bunker, Jr., and Herbert S. Strickler, "The Effect of Mixing Coals on the Ash Fusion Temperature of the Mixture," *Cooperative Bulletin 62*, Carnegie Institute of Technology (1934).

[9] A. W. Gauger, "Coal and Its Mineral Matter," *Bulletin 28*, Mineral Industries Experiment Station, The Pennsylvania State College (1936).

[10] C. G. Ball, *Report of Investigation No. 33*, Illinois Geological Survey (1935); G. Thiessen, C. G. Ball and P. E. Grotts, *Industrial and Engineering Chemistry*, Vol. 28, p. 355 (1936).

forming minerals not only vary from seam to seam but frequently may vary within the same mine. In fact, the only conclusion that the author is prepared to draw at the present moment on the basis of examination of several hundred ash analyses is that there is a tendency for lime to exist in increasingly larger amounts as one proceeds to the south and west from the eastern edge of the Pennsylvania bituminous coal area. In other words, in general, western Pennsylvania, West Virginia and Illinois coals have larger percentages of compounds of calcium than do the coals of central Pennsylvania.

Morrow and Proctor[11] have also shown the great variation that may exist in the ash composition of the different types of coal (bright coal, dull coal, fusain), as well as in the analysis of the ash from the different fractions separated at specific gravities of 1.60, 2.00, and 2.60. Their figures for four different coals are reproduced in Table I.

The fourth factor relates to the opinion held by some that the intimate mixing which takes place in the preparation of the sample of ash for test is not representative of conditions in the fuel bed and that the test, therefore, cannot be expected to predict accurately the extent of clinkering that will occur when the coal is burned.[12] To a certain extent this may be true, but in modern industrial practice, screening of stoker coal, cleaning to remove large aggregates of ash-forming minerals, and agitation of the fuel bed result in a fairly uniform distribution of the minerals and an intimacy of mixture approximating that of the ash softening temperature test. Rather must the failure be ascribed to the different chemical compositions which result in the same ash softening temperatures but which may result in slags of widely different fluid properties. Thus, silica and alumina may be looked upon as the refractories in the ash, whereas the predominant fluxing materials are lime and iron oxide. Starting with the same refractory composition, let us say kaolinite, it is possible to produce mixtures of the same softening temperature by adding iron oxide alone, by adding lime alone, or by adding mixtures of the two. Naturally, a mixture of kaolin and iron oxide with a softening temperature of 2500 F. cannot be expected to respond in a fuel bed in the same way in which a mixture of kaolin and lime of the same softening temperature will behave.

Some engineers are of the opinion that determinations of ash softening temperatures by the laboratory procedure are often deceptive because there may be chemical reactions in the progress of the fuel through the operating furnace which may change the fusing characteristics materially. These engineers suggest that more informative results may be obtained by collecting ash samples from similar types of equipment when burning the coal under investigation and making ash softening temperature measurements on them. Such suggestions serve to emphasize the point that factors other than ash softening temperature as now determined are of great importance in clinker formation.

HOW CLINKERS ARE FORMED

At this time it seems desirable to attempt to visualize the process through which the mineral matter in coal passes during combustion. In the case of bituminous coal, the first step involves the formation of coke, accompanied by such changes as loss of volatile matter, sulfur, carbon dioxide, and water of

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[11] J. B. Morrow and C. P. Proctor, "Variables in Coal Sampling," *Proceedings*, Am. Inst. Mining and Metallurgical Engrs., Vol. 116, p. 249 (1936).

[12] Ralph A. Sherman and E. R. Kaiser "Clinker Formation in Small Underfeed Stokers," *Information Bulletin 1*, Bituminous Coal Research, Inc., January, 1936, p. 2.

hydration of certain minerals. From that point on, the process is not unlike that occurring in the combustion of anthracite. This process has been observed in our laboratories under controlled conditions. If the lump is not disturbed, the combustible matter burns away, leaving the ash more or less in place but accompanied by a certain amount of shrinkage. The infusible components remain as flaky or fluffy ash; the fusible components sinter or melt into globules, depending upon the temperature-composition relations. This material finally works its way down to the grates, during which process the different mineral components of the ash have an opportunity of coming into a more intimate contact. If any component fuses completely, it will flow depending upon its viscosity and may entrap other ash components as well as unburned combustible material. Reactions may take place resulting in more fluid systems such as eutectics. Agitation of the fuel bed aids in the movement of the individual ash particles, making coalescence of fused masses and reaction more easily accomplished.

The important factors at this stage are temperature and properties of the slag. If the individual globules of slag are fluid so that they reach the grate area rapidly, and if the temperature on the grates is low enough so that they solidify rapidly (this also presupposes that the slag has a large temperature coefficient of viscosity), then a loose aggregate of clinker will be formed; this will be handled by the furnace mechanism without difficulty. If, on the other hand, the temperatures on the grates are high relative to the solidification temperatures of the slag globules, the slag will continue to flow, one globule wetting another globule, and so on, until a large dense clinker is formed. This will disturb air flow and will be removed only with difficulty. Such a process gives rise to clinker trouble. Barrett and Taylor,[13] Nicholls,[14] and Bunte,[15] have indicated various ways in which slags may behave upon heating and cooling.

GENERAL SIGNIFICANCE OF ASH SOFTENING TEMPERATURE

All of the above discussion has served two useful purposes. In the first place, it has shown that the formation of troublesome clinker is dependent upon the equipment and firing practice as well as on the inherent characteristics of the coal and ash. It has also shown that the temperature in the fuel bed and on the grates is of great importance. In view of the many factors involved in clinker trouble, no specific statement can be made with reference to the significance of ash softening temperature in all types of coal utilization. It will have one significance with one consumer and another significance with another consumer. This has been recognized in a recent report published by the National Committee on Coal of the National Association of Purchasing Agents.[16]

This committee has rated the various factors as "not important," "of ordinary importance," "of more than ordinary importance," and "very essential." The general conclusions indicate that the ash softening temperature determination is quite an important factor in the different classes of utilization for stationary steam generation under four

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[13] Elliott P. Barrett and James A. Taylor, "A New Method for Studying the Flow Characteristics of Glasses and Slags at Elevated Temperatures," *Journal*, Am. Ceramic Soc., Vol. 19, pp. 39–44 (1936).

[14] Discussion by P. Nicholls of paper by J. J. Brennan "The Fusibility of Coal Ash," *Proceedings*, Seventeenth Fuel Engineers Meeting, Appalachian Coals, Inc., January 11, 1937.

[15] Karl Bunte, "A New Method for the Determination of Melting Processes in Ash," *Proceedings*, Second Internat. Conference Bituminous Coals, Carnegie Institute of Technology, Vol. 2, pp. 231–240 (1928).

[16] Report of Subcommittee VI on Correlation of Scientific Classification with Use Classification, to the Technical Committee on the Classification of Coal sponsored by the American Society for Testing Materials; *Publication No. 21*, Second Edition, published by the National Committee on Coal, National Association of Purchasing Agents (1936).

conditions. These are: excessive load, insufficient grate to heat area, pre-heated air, unfavorable fire-brick conditions. The first three of these are all related to fuel-bed temperatures, and in particular to the temperature on or near the grates. We have already seen that this condition is of great importance. In this connection it should be pointed out that the ash softening temperature is rated less important—dropping from "very essential" to "of more than ordinary importance"—in the case of all of the above four conditions for underfeed stoker equipment when it is provided with water-cooled tuyeres. The committee report gives no clue as to the significance of different values of ash softening temperature, which is not surprising in view of the great complexity of the problem.

LACK OF EXPERIMENTAL DATA

There has been a paucity of experimental study in this field, which is surprising in view of the wide interest in the subject. The most extensive study that has been reported is that of Nicholls and Selvig.[3] Their general conclusions warrant repeating here:

> Based on the scale used in the laboratory tests of the quantity of large clinkers formed, the results show that the tendency to form clinker for the average of all tests on each coal has a fairly general relation to the softening temperature of the ash as determined by the standard method of the American Society for Testing Materials. On the other hand, values for individual coals departed materially from the average curve. Coals having ashes whose fusion temperature was at the low or high end of the softening-temperature scale conformed to the average more closely than did those in the center having softening temperatures of 2400 to 2600 F.
>
> The investigation also showed that no other single value for the fusibility of the ash would give better coordination with the clinkering scale used than does the softening temperature.
>
> The investigation therefore has not proved that the fusibility of the average ash is necessarily an assured measure of the relative clinkering of coals whose ashes have fusion values not greatly different. The clinker formed, as measured by quantity and density, depends on other factors of the coal and on its method of burning.
>
> Although fusibility is a reliable general index, for close comparisons of coals the following factors should be given weight: Size of coal, its caking and coking characteristics as affecting the paths of the ash through the fuel bed, and the proportion of the ash which is segregated in lumps, all these factors being considered with reference to the furnace and fuel bed. This report presents data and discussions that may be of help in making a more intelligent selection, yet the scope of the investigations and the types of burning and fuel bed studied were not broad enough to permit tabulation of values for the above factors and their coordination with each method of burning or service condition. Such exact presentations and conclusions would require the burning of much larger quantities of a variety of coals under the equivalent of plant conditions. This investigation can be considered only as a beginning; the necessity for exact values becomes less as the investment in equipment that will handle clinker in the fuel bed or at its discharge is increased.

Fieldner and Selvig[17] have subdivided coals on the basis of ash softening temperature as follows:

Class 1.—Refractory ash, softening above 2600 F.

Class 2.—Ash of medium fusibility, softening between 2200 and 2600 F.

Class 3.—Easily fusible ash, softening below 2200 F.

These authors considered class 1 as practically non-clinkering; class 3 will

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[17] A. C. Fieldner and W. A. Selvig, "Relation of Ash Composition to the Uses of Coal," *Transactions*, Am. Inst. Mining and Metallurgical Engrs.

form considerable clinker; the clinkering characteristics of class 2 depend upon the furnace temperature, the kind of stoker, and the distribution of the ash-forming constituents in the coal. It will be shown later that such generalizations may be upset to a surprising degree by modifications in combustion equipment and firing practice.

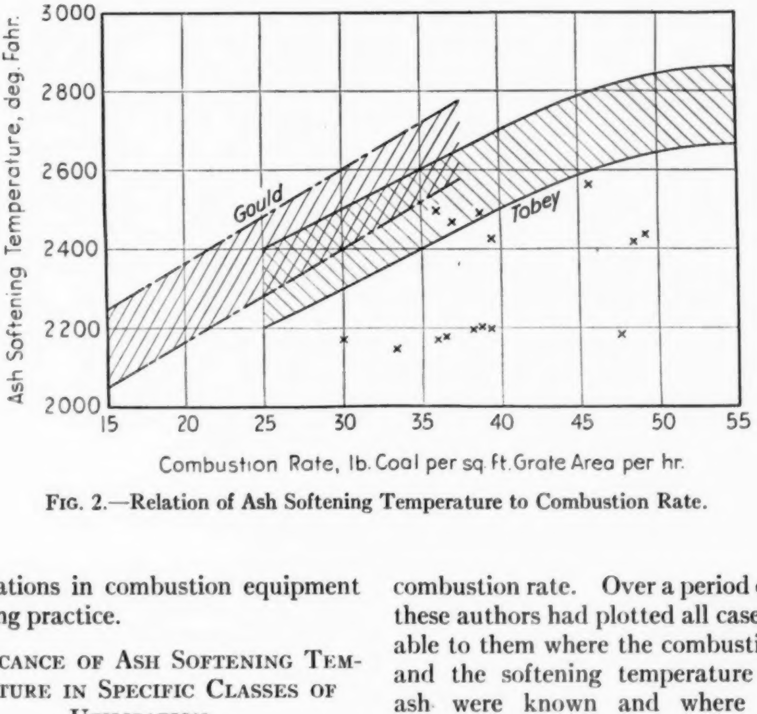

FIG. 2.—Relation of Ash Softening Temperature to Combustion Rate.

SIGNIFICANCE OF ASH SOFTENING TEMPERATURE IN SPECIFIC CLASSES OF UTILIZATION

Having established the importance of factors other than the ash softening temperature, the balance of the paper will sum up the status of the problem in so far as specific classes of uses are concerned.

Underfeed Stoker.—Frequent attempts have been made to relate combustion rates in the burning of coal to the ash softening temperature and clinker formation. The experience with such attempts has served to emphasize that many factors in addition to the softening temperature play equally important roles in the formation of clinkers. In 1929, Gould and Henderson, in a paper entitled "The Recent Trend in Coal Burning Methods,"[18] before the Society of Gas Lighting, presented a chart relating the ash softening temperature to the combustion rate. Over a period of years these authors had plotted all cases available to them where the combustion rate and the softening temperature of the ash were known and where clinker trouble was either definitely reported or known not to have occurred. The points fell within a band as shown in Fig. 2. It was the experience of these authors that, within this band, clinker trouble would almost invariably be encountered at or below the lower limit and practically never at or above the upper limit. Between the two limits, the probability of escaping clinker trouble is greatly diminished the nearer the upper boundary is approached, with

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[18] Private communication.

better than an even chance above the center line of the zone shown on the chart. The data were derived from industrial steam plants of 1000 to 5000 b.hp., a good proportion of which were equipped with underfeed stokers. They were operated with about 12 per cent carbon dioxide and used coal from the northern Appalachian region in run-of-mine or slack size. Gould has the following to say about the interpretation of this chart[18]:

> The validity of this curve depends upon the coal you are using and upon the plant conditions encountered. Introducing a large amount of excess air, which would reduce the carbon dioxide to 6 or 7 per cent, will make an appreciable difference in the clinker formation and permit the use of a lower fusing point coal, but of course at the expense of a substantial loss in efficiency. A refractory arch, such as you have with a Dutch oven with very little of the fire exposed to the boiler tubes, will throw the results over on the other side and clinker trouble would be experienced at lower combustion rates than are indicated on this chart.
>
> The size and coking characteristics of the coal are evidently factors which modify this typical relationship. It would appear, although I do not know of any experimental data which definitely show it, that, other things remaining constant, the tendency to form clinkers will increase as the density of the fuel bed increases. A higher combustion rate can therefore be obtained without clinker trouble with a given fusing point with coal which has been screened to exclude the fines.
>
> It has also been our experience that middle western coals, that is, those from Illinois particularly, do not conform in practice to the relationship shown on the chart. For some reason, which we have been unable to explain, with a given combustion rate, the Illinois coal will not cause clinker trouble, with a fusing point of at least 200 deg. Fahr. lower than coals of similar size, used on similar equipment and under similar conditions from the northern Appalachian fields.

More recently Tobey[18] has prepared a similar chart (Fig. 2) based on the average values taken from data obtained from four different sources (submitted by Rowan, Gould, Williams, and Tobey). The majority of the plants from which performance data were taken burn coal from the eastern and southern fields, including both high- and low-volatile coals. Tobey calls attention to:

> one characteristic of the chart—that is, it is straight, up to the 40-lb. rate. The fact that the curve breaks from a straight line from that point upward is apparently due to an inherent characteristic of the underfeed stoker; that the overfeed section of the stoker (above which the highest furnace temperatures are found) loads up faster than the underfeed section; that is, it reaches its maximum capacity earlier than the underfeed section and much of the additional increase in rating is taken by the underfeed section. For this reason, temperatures do not increase above the overfeed section as rapidly as they did at the lower burning rates.

Generalizations such as these represent the statistical experience of the engineers who make them and are undoubtedly applicable to the conditions in the plants represented. Because of the many factors which have a bearing on the problem, the experience of other engineers observing quite different power plants may be quite different. The author has gathered from several engineers data which are at complete variance with the curves of Gould and Tobey. These are plotted as separate points in Fig. 2. It appears that those coals which do not coke strongly form one exception. Furnace design, firing practice and ash composition are also among the factors which affect the formation of clinker.

Bennett and Herbeck[19] describe a

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[19] J. S. Bennett and C. J. Herbeck, "Design and Development of a Water-cooled Underfeed Stoker," *Mechanical Engineering*, December 1935, pp. 761–765.

water-cooled underfeed stoker which successfully burns a coal of ash softening temperature of 1900 F. at a rate of 52 lb. per sq. ft. of air admitting surface per hour. Careful air distribution may accomplish a similar result because of the cooling effect on the grates. Rosin[20] in Germany has concluded that control of temperature in the fuel bed and in the gases of the combustion chamber, control of atmosphere and control of motion of fluid slag offer the weapons of firing technique against the clinker problem. By certain modifications in design providing for cooling along the side walls, at the tuyeres, and on the extension grates, a lowering of the temperature of the coke amounting to as much as 1000 F. and more is accomplished. The effect of this on clinkering is, of course, obvious.

Unquestionably many factors are of importance in the formation of troublesome clinkers in underfeed stoker practice. The necessity of having to build up a fire very quickly from a banked condition will often cause a black, vitreous clinker. The amount of ash is also of importance. Frequently, it has been reported that, with a high-ash coal of low ash softening temperature, no clinker trouble is experienced even at high rates of combustion (50 lb. per sq. ft. per hr.), whereas with a coal having an ash content of 6.7 per cent it was impossible to reach that burning rate. The reason for this may be the insulating effect of the large volume of ash formed in burning the high-ash coal. It must not be concluded that this is always the case, for there are instances when the reverse is true, all of which is further evidence that the formation of troublesome clinker is the resultant effect of a variety of causes.

Pulverized Coal.—In the case of pulverized coal for purposes of steam generation the trouble experienced is mainly due to slag formation on the water tubes and to the slagging of the refractories in the furnace chamber. Pulverized coal installations are of two types: namely, the dry bottom, and the slag-tap types. At first glance, it appears that an ideal solution of the entire clinker problem is presented by these two types of installations. Whereas coals with high ash softening temperature might command a premium in stoker or dry-bottom equipment, they would be useless in the slag-tap installation. Just the reverse might be expected to be true in the case of the coals with low ash softening temperature. As a matter of fact, this is a correct statement of the situation only within certain limits. While it is true that the ash with high softening temperature is useless in slag-tap furnaces, it is not true that all coals with low ash softening temperature behave similarly. Bailey and Hardgrove[21] state that "the slag-tap furnace can be successfully used for all coals where the ash has a fusing temperature below 2500 F."

McCausland[22] cites a case where one coal with an ash softening temperature of 2500 F. gave highly satisfactory results at Deepwater, whereas another with an ash softening temperature of 2330 F. caused considerable damage to the bottoms and unusual clinkering on the tubes. This presents an interesting case worthy of further elaboration. The slag wells were constructed of refractories and apparently could not withstand the expansion and contraction created by the cooling effects of the slag at the various boiler ratings. With a

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[20] P. Rosin, "The Ash Problem in Combustion," *Zehnte Technische Tagung* des Deutschen Braunkohlen-Industrie-Vereins, E.V., April, 1931, *Braunkohle*, Vol. 31, pp., 628–644 (1931).

[21] E. G Bailey and R. M. Hardgrove, "The Slag-Tap Furnace and Its Effect upon the Selection of Coal for Burning in Pulverized Form," *Proceedings*, Third Internat. Conference Bituminous Coals, Carnegie Institute of Technology, Vol. 2, pp. 349–369 (1931).

[22] J. R. McCausland, "Selection and Utilization of Fuels," *Proceedings*, Nat. District Heating Assn., Vol. 23, pp. 100–125 (1932).

constant run of the same coal these cracks would frequently fill up with slag and be temporarily sealed. However, if a change of coal was made, and particularly if the coal was high in iron and sulfur and created a new fluxing action, it would immediately purge the whole slag pit, open up the cracks that had temporarily been sealed, and leak out on the floor below.

The matter of tube and wall slagging while considered for pulverized-fuel firing presents the same problem in all types of combustion when high air velocities are used. It is evident that any prediction of slag accumulation on tubes which is based solely on a study of coal ash as a whole must be erroneous. While in grate firing the bulk of the ash remains in the bed and the various components may be brought into more or less intimate contact with each other, the condition along the walls and among the tubes in the combustion space is quite different. Since only selected portions of the ash may arrive at such points, the behavior of individual components becomes of great importance. Sherman[23] has concluded from his studies that the "selective deposition of the material on boiler tubes shows that the possibility of slagging of the tubes cannot be predicted accurately from the composition and softening temperature of the ash."

TABLE II.

Wall Construction	Heat Release, B.t.u. per cu. ft. per hr.			
	Ash Fusion 2200 F. and below	Ash Fusion 2200 to 2400 F.	Ash Fusion 2400 to 2600 F.	Ash Fusion above 2600 F.
Solid walls	never	12 500	15 000	15 000
Air-cooled walls	12 000	15 000	17 500	18 500
Water-cooled bottoms	16 000	17 500	20 000	22 000
Water walls	17 500	22 000	30 000	32 000
Complete water-cooled furnace	19 000	24 000	32 000	36 000

Fettke and Stewart[24] have recently reported on an elaborate investigation of the slagging of refractories by ash from coals of western Pennsylvania. They conclude that the temperature of the refractory surface must exceed the ash softening temperature before erosion of the refractory can occur. However, certain types of refractories show no erosion at temperatures appreciably above the ash softening temperature. "Hence the ash softening temperature as measured by the A.S.T.M. method is not an exact measure of the critical temperature above which slag attack starts."

It is evident that many factors are involved in the successful burning of coal in pulverized fuel installations. Rowan, in a letter to the author, suggests a general relationship between (*a*) the number of B.t.u.'s released per cubic foot of furnace volume per hour, (*b*) the amount, character, and softening temperature of the ash in the coal, and (*c*) the type of furnace in which the coal is burned as presented in Table II.

An excellent discussion of this phase of the subject is contained in the 1936 report of the Combustion Subcommittee of the Prime Movers Committee,[25] from which the following is quoted:

> The furnace section of the unit is possibly the most important element in the design. Satisfactory furnace performance is obtained when the unit will operate without slagging of the convection tube bank, the water walls will withstand the intense heat developed in the process of combustion of fuel, and the carbon loss will be at a minimum. The combination of slag-screen and slag-tap furnace represents the most

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[23] R. A. Sherman, "A Study of Refractories Service Conditions in Boiler Furnaces," *Bulletin 334*, U. S. Bureau of Mines (1931).

[24] C. R. Fettke and W. E. Stewart, "The Slagging Action of Western Pennsylvania Coals on Different Types of Refractories in a Small Pulverized Coal Installation," *Bulletin 73*, Mining and Metallurgical Investigations, Carnegie Institute of Technology (1936).

[25] Report of the Combustion Subcommittee of the Prime Movers Committee, *Publication D-7*, Edison Electric Inst. (1936).

popular current development to improve furnace performance.

Regardless of the type of boiler and the method of pulverized coal firing employed, the temperature in the active zone of combustion will approximate 3000 F. The extent or volume of this zone of high temperature will vary in accordance with the type of heat-absorbing surface used, the volume of the chamber in which active combustion occurs, and the method of firing or type of burner employed.

To maintain the boiler tube bank in a slag-free condition, the temperature of the gases entering the boiler tube bank should approximate the fusion temperature of the ash. Temperatures somewhat higher than the fusion temperature are permissible in some cases, depending on the spacing of the first few rows of the tube bank and the number of vertical rows of tubes installed. It is clear then that for successful operation the heat absorption or the temperature drop from the combustion zone to the boiler tube bank must be substantially the same in all makes of furnaces.

TABLE III.

Company	Date of Installation	Maximum Steaming Rate, lb. per hr.	Maximum Heat Release B.t.u. per cu. ft. per hr.	Satisfactory Tapping, B.t.u. per cu. ft. per hr.	Fusion Point of Ash, deg. Fahr.	Type of Furnace	Maintenance, cents per ton of coal
Appalachian Electric Power Co.	1931	355 000	19 950	14 000	2 800	Water cooled	0.5
Buffalo General Electric Co.	1930-1931	560 000	35 700	6 900	2 100 to 2 500	Water cooled	0.6
Deepwater Operating Co.	1929	320 000	18 200	18 200	2 450	Water cooled	4.8 (4 yr. average)
E. I. du Pont de Nemours and Co.	1936	75 000	25 300	11 800	2 200		
Firestone Tire and Rubber Co.	1935	350 000	24 100	10 300	2 100 to 2 200	Water cooled	
Iowa-Nebraska Light and Power Co.	1935	160 000	31 750	15 880	2 100 to 2 200	Water cooled	
New York Power and Light Corp.	1932	350 000	33 400	23 900	2 300 to 2 400	Water cooled	
Northern Indiana Public Service Co.	1930	345 000	31 800	22 200	2 300	Water cooled	5.6 (Boiler and Furnace)
Ohio Power Co.	1931	320 000	29 000	20 000	1 900 to 2 100	Water cooled	2.0
Philadelphia Electric Co.	1935	600 000	30 000	30 000	2 300	Water cooled	
Potomac Electric Power Co.	1933	375 000	35 000	28 000	2 500 to 2 600	Water cooled	
Public Service Company of Northern Illinois	1930	300 000	26 500	22 100	2 100	Water cooled	
	1931	500 000	27 200	19 000	2 100	Water cooled	0.9
Sinclair Refining Co.:							
Plant A	1930	120 000	30 000	15 000	2 300	Block	2.5
Plant B	1931	150 000	32 000	21 300	2 400	Block	1.8 (coke)
Super Power Company of Illinois	1930	45 000	27 400	16 800	1 950	Water cooled	2.5

Heat transfer rates through water walls by radiation have been fairly well established for the many different types of water cooling construction used by different manufacturers. It therefore becomes a more or less practical design problem to establish the heat absorbing area necessary to produce the proper gas temperature in order to prevent slagging and at the same time to maintain superheat at the proper value. The range of gas temperatures to accomplish these ends, particularly in installations having steam temperatures 900 F. and higher, narrows down to fairly close limits...

The slag-tap furnace as originally designed was for intermittent tapping; however, in some of the most recent installations a continuous-tapping arrangement has been adopted. One company is now converting a furnace from dry-bottom to continuous-tapping design, while another has recently installed a continuous slag-drip furnace to replace stoker-firing equipment. Several operating companies state that the design of burners and method of firing have an important bearing on the ease of tapping the slag. Iron sulfide still seems to be a disturbing element with some slag-tap installations, as brought out in several operating company statements. Three companies report troubles with slag spout wear which has been eliminated by water cooling the slag spout and making the spout liner of special materials.

The committee has gathered data on over-all furnace heat release and furnace maintenance cost for slag-tap boilers from a number of operating companies and two industrial companies. These data are shown in Table III....

The heat liberation per cubic foot of furnace volume per hour has been used to a considerable extent as a measure of operating and design results. This unit, however, merely gives an overall result and a somewhat uncertain relationship between total furnace space and fuel consumed....

In connection with the value of heat-release data, the statement of one manufacturer reports that: "The experience of this company indicates that a better index of furnace performance than the heat release per cubic foot is the heat release per square foot of furnace cooling surface. In small furnaces the available wall area per cubic foot of furnace volume is greater than in large furnaces, hence higher heat releases per cubic foot of furnace volume are possible in small furnaces."

Tables IV and V, which show the relationship between ash softening temperature and maximum furnace heat release in dry-bottom and slag-tap furnaces, are quoted from the 1935 report of the Combustion Subcommittee of the Prime Movers Committee, Edison Electric Institute.[26]

Domestic and Miscellaneous Uses.—Johnson[27] has reported on some very interesting experiments with an 18-in. round boiler of the domestic type using anthracite. A cooling surface, consisting of a water-cooled cylinder in the heart of the burning coal was installed in the boiler. With an anthracite coal having an ash softening temperature of 2300 F., the maximum burning rate (without clinker trouble) was increased from 3.8 lb. per sq. ft. per hr. to 8.4 lb. per sq. ft. per hr. with chestnut size and from 5.0 lb. per sq. ft. per hr. to 10.1 lb. per sq. ft. per hr. using stove size by means of this simple expedient.

Table IV.—Relation of Ash-Fusion Temperatures and Furnace Heat Release for Dry-Ash Removal.

Companies with Dry-Ash Removal	Air Fusion Temperature, deg. Fahr.	Maximum Furnace Heat Release, B.t.u. per cu. ft. per hr.
Cleveland Electric Illuminating Co.	2 200	18 600
The Detroit Edison Co.	2 500	15 000
Edison Electric Illuminating Company of Boston	2 350 to 2 900	28 000
Hartford Electric Light Co.	2 650	30 000
Kansas City Power and Light Co.	1 900	20 000
Milwaukee Electric Railway and Light Co.	2 100	18 000
Penna. Water and Power Co.	2 600	20 000
Public Service Electric and Gas Co.	2 400 to 2 600	25 000
West Penn Electric Co.	2 350	18 070

Table V.—Relation of Ash-Fusion Temperatures and Furnace Heat Release for Molten-Ash Removal.

Companies with Molten-Ash Removal	Ash Fusion Temperature, deg. Fahr.	Furnace Heat Release, B.t.u. per cu. ft. per hr.	
		Minimum for Tapping	Maximum
Buffalo General Electric Co.	2 100 to 2 500	6 900	35 700
Deepwater Operating Co.	2 260 to 2 420		20 300
Ohio Edison Co.	2 000 to 2 500	10 000	25 000
Oklahoma Gas and Electric Co.	2 000 to 2 200	14 400	25 000
Public Service of Colorado	2 250 to 2 550	22 000	
West Penn Electric Co.	2 350	17 070	24 800

In the case of the blue gas generator, Warnick[28] has made a careful study of the causes of clinker formation. He lists the character of the ash, the fusing temperature of the fuel bed, the length of the operating cycle, and the rate of

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[26] Report of the Combustion Subcommittee of the Prime Movers Committee, Edison Electric Institute, *Publication C-6*, 1935.

[27] Allen Johnson, "Furnace Conditions as Related to the Formation of Clinker," *Report No. 2326*, Anthracite Institute Laboratory, October 22, 1936.

[28] J. H. Warnick, "Cleaning of Generator Fires and Progress Towards Clinker Prevention," *Proceedings*, Am. Gas. Assn., pp. 816–819 (1924).

blasting as of importance in this respect. According to Warnick:

> Clinker troubles are at a minimum when the fusing temperature of the ash is between 2300 and 2500 F. As the ash fusing temperature is lower or higher than the limits stated above the troubles develop in the formation of clinker on the grates or on the walls of the generator lining.

This is readily understood if one keeps in mind the mechanism of operation of the blue gas generator. The alternate blasting and steaming of the fuel causes a temperature cycle that reaches a maximum at the end of the blast and a minimum at the end of the run. Since clinker is formed by solidification of a fused slag, the coals with high ash softening temperature will form clinker high up on the generator lining. Those with low ash softening temperature will yield a slag that remains fluid on the grates and forms the very troublesome pancake clinker. On the other hand those with medium ash softening temperature will fuse in the hot zone and solidify on the way down to the grates.

The present practices in water-gas manufacture, which include with the alternate blasting and steaming cycles a split-up of the steaming cycle into up and down steaming, can be used to vary the position of the clinker. To illustrate this with the same fuel, if we were blasting and using steam in the up direction our hot zone would be toward the upper part of the fuel bed in the generator. If we were blasting and using steam in the down direction, our hot zone would be in the lower part of the fuel bed in the generator. By varying the time of the up and down steam runs, we can control the position of the hot zone and to a considerable extent the clinker line which assists considerably in cleaning the generator.

Warnick has also pointed out that the fusing temperature of the ash bears a definite relation to the quantity of air that can be blasted through the fire during a blow. With a softening temperature as low as 2200 F. the blasting rate per square feet of grate per blow should not exceed 350 to 400 cu. ft. As the softening temperature approaches 2600 F. the blasting rate can be increased to a figure as high as 500 to 550 cu. ft. per sq. ft. grate area per blow.

The effect of the coking process on the ash softening temperature has been studied by several laboratories with a general conclusion that the softening temperature of the ash in the coke is, within the limits of precision of the test, the same as that of the original coal.

If such investigations are made by using the standard methods of determining ash fusions, the results should show within the limits of the precision of the test the same ash fusion as that of the original coal. In the process of coking the oxides in the ash may have been reduced ashes and therefore the ash should be the same as the original coal ash which is also reoxidized.

COMPOSITION OF COAL ASH

For a long time the problem of relating the ash analysis to the ash softening temperature in such a way as to make it possible to calculate the latter from the former was considered to be too complex for solution. However, within the past several years at least two methods of calculating ash softening temperature from ash analysis have been worked out. There are nine components in coal ash that influence the softening temperature to varying degrees. These are SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO, K_2O, Na_2O, P_2O_5, and TiO_2. Of these, it may be said that the effects of TiO_2 and P_2O_5 are negligible; other factors being equal, the higher the ratio of Al_2O_3 to SiO_2, the higher will be the softening temperature; the higher the percentage of alkalies, alkaline earths and iron oxide, the lower will be the ash softening temperature.

The presence of quantities of iron oxide in the ash in excess of 20 per cent is accepted by some technologists as being responsible for serious clinker trouble. Thus McCausland[29] believes that iron oxide in the case of slag-tap furnaces is likely to be detrimental and Barkley[29] refers to the use of the following clause in certain Government specifications for the purchase of coal:

> The ash softening temperature limits given in Schedule 1 are for coals having not more than 20 per cent iron oxide in the ash. The right is reserved to reject bids offering coals having higher than 20 per cent iron oxide if the ash fusion temperature is less than 2700 F.

It must be emphasized that exceptions occur. McCausland[29] has written of such a case:

> Recently we tested a medium volatile coal from Central Pennsylvania in which the iron oxide was approximately 28 per cent, the volatile 18.59 per cent and the ash 10.60 per cent. This particular coal seemed to have a purging action on the boiler, not only cleaning off all the old slag but continuing to free itself. Of course it required a longer tapping period and some extended operation of the mills due to the increased ash. Whether after a longer duration of time we would find that the fly ash from this coal deposited in another part of the boiler and gave trouble I am not in a position to say but during the week's operation it completely purged the boiler of all clinker formation. The fluid on this coal according to A.S.T.M. standards was 2550 F., whereas in the initial it was 2170 F., which again goes to prove that the fusion characteristics of the ash are not always the criterion.

It should be emphasized that Barkley was one of the first to attempt a correlation of the ash softening temperature with ash action in steam generation. His conclusions published as ***Report of Investigation No. 2630*** in August, 1924, by the U. S. Bureau of Mines are equally applicable to the situation today.

For a detailed discussion of the properties of slags and the effect of composition on these properties the reader is referred to two papers by Nicholls and Reid.[30]

Certain special-purpose coals may require the presence or absence of certain mineral components in utilization. The most important of these are calcium oxide which is a desirable component in the case of the cement industry, and phosphorus and sulfur which must be kept at a minimum in coking coals destined for the iron and steel industry.

EPILOGUE

Completion of this manuscript leaves the author with an air of uncertainty as to just what has been accomplished thereby. Thousands of ash softening temperature determinations have been made and are being made. What is the significance of all this laboratory work? Hundreds of contracts include an ash softening temperature specification. Why? Thousands of consumers with various types of equipment show a preference for coals with ash softening temperature over 2700 F. Is there any logic in such a situation or are many of these consumers in the same situation as was early reported by the Committee on Use Classification of Coal for Stationary Steam Generation as indicated by the following quotation:[31]

> On the other hand there are numerous specific cases which demonstrate apparent lack of knowledge of the relationship between coal characteristics and plant performance. Several plants report clinkering as a major consideration in the choice of

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[29] **Private communication.**

[30] P. Nicholls and W. T. Reid, "Fluxing of Ashes and Slags as Related to the Slagging-Type Furnace," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 54, pp. 167–190 (1932); "Slags from Slag Tap Furnaces and Their Properties," Vol. 56, pp. 447–465 (1934).

[31] **Thomas W. Harriss, Jr., "Use Classification of Coal for Steam Generation," presented at the New York meeting of Am. Inst. Mining and Metallurgical Engrs., February, 1933.**

coal, and also definitely state that the fusing point is either "not known" or "not determined." A still larger number, though of course a small minority of the whole number, report clinkering of major importance, and at the same time report the use of coal having a fusing point in the neighborhood of 2300 F., though these plants were all situated in an area where much higher fusing point coal is easily obtainable. Three plants, all operated by the same company, all indicating clinkering as important, and all using anthracite, report the fusing point as 2300 F., although it would take a very careful search of the anthracite fields to find any coal having a fusing point anywhere as low as that.

Another plant reports clinkering as its first consideration, next to cost, and gives 2375 F. as the fusing point of its first choice of coal, and 2800 F. for its second choice.

One conclusion that might be reached is that expressed by Barkley in a letter on this subject:

> I find the use of the ash softening temperature very helpful in purchasing coals for the hundreds of plants in the Government service. Although not absolutely accurate it is sufficiently so to be used as a practical guide, particularly in eliminating coals from consideration. Its use must, of course, be accompanied with considerable judgment in each individual case. I have not found it expedient to make any studies in connection with coal use on any of the other determinations, such as flow interval, etc. I understand that certain individual plants have found it desirable to consider along with the ash softening temperature, the initial deformation temperature. One of the first things I wish to know about any coal is its ash softening temperature. I consider it a very important laboratory determination, even if its use in anticipating clinker or slagging troubles in practice is not a simple 1 + 1 proposition. Experience builds up knowledge how to make use of the factor.

The author prefers however to sum the matter up in another way. It is evident that clinker formation is a resultant of many contributory causes. The coal, by virtue of its mineral matter, supplies the raw material from which clinker is formed. The process of formation is, however, dependent upon factors largely independent of the mineral matter in the coal. The process is one of physical and chemical reaction in a complex system of silicates, and, like all chemical reactions, is aided and abetted by heat and agitation. Time of course is another factor, but it is closely related to heat and agitation.

Clinker trouble is an ailment that affects the consumer, and frequently his natural reaction is to blame the coal. This is not surprising in view of the fact that he is often able to switch to another coal, and quick-witted salesmen are always ready to offer another coal "guaranteed" not to clinker. A state of mind thus grew up which attached clinker trouble to some property of the coal, and this was aided and abetted by the fuel technologist who said: "Here is a property called fusibility. It is responsible for clinker formation." There has thus been a tendency among some consumers to overlook the many other factors that are necessary to clinker formation.

The author believes that the importance of ash fusibility as measured by the standard A.S.T.M. ash softening temperature test has been over emphasized. Had there been no coal with highly refractory ash, the problems incident to ash disposal (including clinkering, etc.) would have been solved by equipment design and firing practice. Since the reserves of coal with highly refractory ash are not large, these problems will ultimately have to be solved in that manner.

In the meantime it is evident that the present ash softening temperature method is only a qualitative index of the probable severity of clinker formation;

consequently, a search will continue for methods which offer promise of being better indices. Several such interesting developments are under way, but, since this paper is concerned only with the significance of the present standard methods, a discussion of these is beyond its scope.

ADDITIONAL REFERENCES

This paper does not pretend to include a complete bibliography on the subject. In addition to those already cited, the following references have also been consulted:

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DISCUSSION

MR. J. F. BARKLEY[1] (*presented in written form*).[2]—Mr. Gauger indicates that the ash softening temperature of a coal is not the index of clinkering tendencies that many may have assumed it to be. The ash fusion of a coal as determined in a laboratory shows what happens to the ash of a coal under certain conditions. Observation in the plant will show that this laboratory determination is not the only factor involved in clinkering. It is, however, an important factor that should be given consideration. How and to what extent it should be given consideration depends upon the individual case.

As a simple example, assume a plant with underfeed stokers operating under certain required conditions. From experience it is learned that when a $1\frac{1}{4}$-in. slack coal from a given seam is used, an ash softening temperature of say 2500 F. gives satisfactory conditions from a clinkering standpoint. We will further assume that in handling the coal in the plant, a great deal of segregation in size takes place. It might be found that satisfactory conditions from a clinkering standpoint could be had with coals of a lower ash softening temperature if:

1. Segregation of sizes could be prevented.
2. A different size coal were used.
3. Coal from a different mine or seam were used.
4. Coal with a lower ash content were used.
5. Various adjustments in the stoker were made.
6. Better stoker operators provided.
7. Lower CO_2 were carried.
8. More boilers were put on the line.
9. Certain changes of a minor or major nature in the equipment were made.

Such a list could be extended to cover many factors. When such changes are made that are economically and practically feasible, a status is usually reached where the ash softening temperature factor has considerable significance. The actual temperature might not be the same for coals from different seams or of different sizes; but experience has shown that decisions can be made more readily and at less cost if the ash softening temperature is known. One case comes to mind where no cognizance was taken of the ash softening temperature in searching for a satisfactory coal. Some forty coals were tried out at much expense and trouble. Three were found to be satisfactory. Later, the case as a whole, including the coal analyses and the ash fusion temperature, was referred to an experienced fuel engineer. Without knowing the results of the trials, he picked out five coals that he would have recommended for trial, all three of the satisfactory ones being included.

Any attempt to make an all-inclusive correlation of the ash softening with probabilities of clinkering should be in a form that is simple and readily applicable to any individual case. Many studying this problem have long desired that accurate and complete observa-

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[1] Supervising Engineer, Fuel Economy Service Station, U. S. Bureau of Mines, Washington, D. C.

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tions be made at many individual plants of all factors related to clinker formation. It is conceivable that all such observations might be summed up in the form of an all-inclusive correlation, but such a formula, if it could be devised, would be so cumbersome and complicated that it would be of value only to a few. Attempts at all-inclusive correlation may lead one into the position of the confused viewpoint of the man who could not see the forest because of the trees.

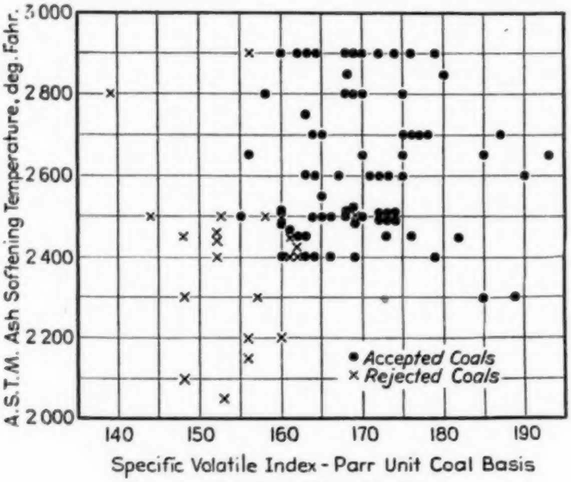

FIG. 1.—Results of Burning Tests.

Some coal users, at present, are searching for other relatively simple laboratory analyses that may fit their particular needs in anticipating clinkering difficulties somewhat better than does the standard ash softening temperature. It seems appropriate to mention a paper along these lines by A. H. Moody and D. D. Langan, of the United Electric Light and Power Co., entitled "Fusion Characteristics of Fractionated Coal Ashes," published in *Combustion*, October, 1933. This paper describes investigations of the relation of certain clinkering actions on underfeed stokers with the tendency of the ash to separate, part leaving the fuel bed with the products of combustion and part remaining.

Whatever might be the general technical significance of the ash softening temperature to clinkering tendencies on the basis of some chosen scheme of correlation, its economic significance is attested by the demand over a period of years for such analyses.

MR. A. W. THORSON[3] (*presented in written form*).—In The Detroit Edison Co. we analyze each of the coals we burn for ash softening temperature by the A.S.T.M. method, for use as a guide in our coal selection.

As an operating routine we also determine the ash softening temperature of a composite sample representing each month's supply of coal to each power house.

Comparison of these data with clinkering and slagging in the boiler furnaces has failed to show correlation within the limits of variation we normally encounter (2400 to 2900 F. ash softening temperature). For example, a plant may have had no clinkering difficulties

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[3] The Detroit Edison Co., Detroit, Mich.

one month when the average ash softening temperature was 2450 F., and may have encountered some trouble the following month when the average ash softening temperature was 2550 F.

We attribute this lack of correlation to the fact that we are burning a mixture of coals from many seams and mines, and the proportions of the mixture vary from day to day. The average ash softening temperature from a composite sample, therefore, is not a reliable index.

We have been able to show a much better correlation when burning individual coals. Results of burning tests are shown in the accompanying Fig. 1. These tests of proposed coals are made with two purposes in mind: first, to determine the tendency to clinker, and second, to determine whether the required maximum combustion rate can be maintained. On the basis of these results approximately 2500 F. was selected as the minimum ash softening temperature permissible for our stoker-fired plants. The two high ash fusion coals were rejected because they lacked sufficient coking properties. When attempting to maintain the required maximum combustion rate, an excessive amount of coke was blown from the fuel bed into the ash pit, resulting in intolerable ash pit loss.

The writer wishes to comment on the author's Fig. 2, combustion rate *versus* ash softening temperature. Two more points, at variance with the bands, are offered. Successful operation has been maintained at Delray with coal of 2500 F. ash softening temperature and at Conners Creek with coal of 2600 F. ash softening temperature, both at combustion rates above 55 lb. per sq. ft. per hr.

It is believed that combustion air temperature and size of stoker, neither of which was mentioned, also are factors in the relation. Preheated air raises the fuel bed temperature, thus moving the band upward with increasing air temperature. As the size of stoker increases, the fuel bed becomes less uniform. Small zones at high rates of combustion will cause clinker even though the average ash softening temperature, as measured by the cone test, appears reasonable for the average combustion rate. This factor also moves the band upward as stoker size increases. Zoned-air control, however, minimizes this latter factor.

It is quite probable that the importance of coal ash fusibility has been overemphasized by many. It is only one of many properties to be considered in coal selection. It is useful, however, within its limitations, and we should not care to dispense with it unless a more reliable index were available.

MR. J. S. BENNETT, 3D.[4]—Mr. Thorson has stated that the practical coal burning rates obtained when using zoned or "metered" air control with multiple retort underfeed stokers are much greater than those indicated by Fig. 2 of the author's paper. This is due, undoubtedly, to the fact that a rate of 45 lb. per sq. ft. per hr. in Fig. 2 is an average rate and represents a maximum rate in spots of say 75 lb. per sq. ft. per hr., while other sections have a rate of, say, only 20 or 30 lb. per sq. ft. Sectionalized air control forces the sluggish portions to do their share of the work, and raises the average combustion rate without increasing the maximum rate, and consequently without increased difficulties. Any method that promotes the uniformity of coal feed, of coal distribution, or of air flow, increases the permissible coal burning rate. The writer witnessed an 8-hr. run recently where all three of these factors have been considered and although zoned air con-

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[4] Mechanical Engineer, American Engineering Co., Philadelphia, Pa.

trol was not employed, an entirely satisfactory rate of 80 lb. per sq. ft. per hr. was maintained when the air for combustion was supplied at a temperature of 560 F. According to Fig. 2 the highest permissible rate would be between 45 and 50 lb. per sq. ft. per hr. Care should be taken to remember that the data given in this figure represent past practice but do not represent a principle that need limit future performance.

MR. P. NICHOLLS[5] *(presented in written form)*.[6]—Mr. Gauger's interesting paper rather fails to reach the definition called for by the title. He handicaps himself at the start in accepting the statement that "the ash softening temperature is intended to be an index of clinker trouble," and as a result he finishes his review rather pessimistically in that he "believes the importance of ash fusibility . . . has been overemphasized." Is it not nearer the truth to say that we have abused these measures and have attempted to give them a significance beyond that warranted; we have hoped that they would explain everything that is directly or indirectly associated with the ash in the utilization of the fuel containing it. We might almost as well expect the tensile strength of metals to predict what will happen to structures in which they are used without taking into account the design of the structure and the forces applied thereto. However inadequate the ash fusion temperatures may be, at least they have filled a gap and we would be much handicapped if this measure were not available. We would be in much the same condition as we now are because of the lack of standard measures of the caking properties of coals and probably worse off, because variations in the caking properties of a given coal are less than may be that of the ash of a given shipment from one mine.

The author does not discuss the meaning of the cone fusion temperatures nor the justification for assuming that meaning, which should be the first consideration. The cone data go no further than attempting to define the temperatures at which finely crushed samples of the average of the whole of the ash will have similar degrees of fusion, which we might hope to mean equal viscosities. However, other factors than the viscosity enter into the deformation of the cone, namely, the surface tension of the slag, the restraint of the base, and the reaction between the slag and the base; the last action will materially affect the observation which gives fluid temperature. It has not been shown how closely the fusion temperatures satisfy the assumption of the same viscosity for all compositions of the ash at the softening and at the fluid temperatures. That the viscosities may not be the same is indicated by the fact that the temperatures as determined depend on the rate of heating and may have other values if the ash is melted, reground, and then used in the cone.

It is apparent that there is no justification for claiming that the cone method of determining the melting properties of ashes is perfect. The ideal would be to use a method which would give a plot of viscosity against temperature, but such determinations involve difficulties and are expensive, although the expense of determining two points for viscosities corresponding to the softening and fluid temperatures would not be much less than determining enough points for a complete curve. It is questionable whether such expense would be warranted because of the inability in most application to fix the values for the other factors which affect the forms

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[5] Supervising Engineer, Fuels Section, U. S. Bureau of Mines, Pittsburgh Experiment Station, Pittsburgh, Pa.

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the ash takes in the use the fuel is put to. Also, it cannot be claimed that we have attempted to make the full use of the fusion temperatures, because but rarely do we try to use any but the softening temperature; a consideration of the fluid temperature and the fluid interval can sometimes be of advantage.

A large interval between the initial and softening temperatures indicates that there are some constituents in the ash which are more fusible than the average ash which takes longer to combine; a long interval will occur if there is much free alumina and the intrinsic ash is low fusion. The interval between the softening and the fluid temperatures is the more useful, especially if the softening temperature is low. This interval gives some measure of the composition of the ash, but not many of the cone data have been worked up as we have used a different method in most of our work on slags. In one instance six ashes all having a softening temperature of about 2000 F. were picked out and the fluid interval plotted against the percentage of ($CaO + MgO$); this plot gave a straight line within the accuracy of the cone data. The ash at one extreme had 45 per cent Fe_2O_3, 1.8 per cent ($CaO + MgO$), and a fluid interval of 370 deg. Fahr.; at the other end the interval was 110 deg. Fahr. for 15 Fe_2O_3 and 17 ($CaO + MgO$). This knowledge is very useful if one knows nothing about the composition of the ash. There is a limit to such interpretation of the cone data because even with the same chemical analysis of the coal ash the melting of the cone depends on the mineral forms in its make-up.

The author uses the terms "tendency to clinker" and "trouble" more or less interchangeably, but they are by no means synonymous. Tendency to clinker is more nearly related to the fusion values and is the first step; trouble is an indefinite statement which operators but rarely define, and it usually involves more factors than the former term. The fusion temperature can be a good measure of the tendency to clinker if the conditions to which the ash is subjected approach those of the cone test, that is, if the coal pieces are very small so that the ash is well mixed. Such an instance is the similar burnings of coke made from small coal.

If the pieces of extraneous ash are large and not well mixed, the ash fusions will not be true relative measures of the tendency to clinker, unless the method of burning be such as to maintain the released ash in the high-temperature zones for a long enough time for the more refractory lumps to be absorbed into the slag from the more fusible ash. The author does not emphasize the importance of the time of exposure factor sufficiently; an Illinois coal of large size may often not show a tendency to clinker equivalent to its fusion temperature, partly because of lack of time for the ash to agglomerate, but mainly because the average temperature of the fuel bed is lower (see p. 39 of footnote reference 3 of the paper). This explains the quotation from Mr. Gould of the less clinkering that he observed with Illinois as compared with Appalachian coals. It is doubtful if we would be much better off if we had complete viscosity curves; they would be useful in instances where the imposed conditions are relatively simple, such as in a slag-tap furnace, or for detail studies, but they are not required nor could the data be effectively used in most uses of coals, although that is no reason why we should rest satisfied with the present standard.

No measures of the properties of the average ash, however complete, are sufficient by themselves for the prediction of clinkering troubles; they could not relieve us of the necessity of evaluating the parts other factors play if we want to explain—not to mention predict—the ash factor. The action and effects of these other factors are often very complex and explaining is a great deal simpler than predicting.

MR. R. M. HARDGROVE[7] (*presented in written form*).—Previous discussions have pointed out that initial and fluid temperatures were not mentioned in the paper and that they should be considered. Our comments are on this same phase of the subject.

The consideration of softening temperature alone may be justified when considering clinkering in solid fuel beds, either hand fired or stokers, but in pulverized coal furnaces both softening and fluid temperatures and the spread between them have a significance that cannot be ignored.

It is well known that slag in the fluid state will not build up in objectionable quantities and in water-cooled furnaces can be handled without difficulty. Also, ash in the dry state is not troublesome. In between the fluid and dry state is a sticky range which is most troublesome.

A coal having a narrow spread between softening and fluid temperature would be expected to change from the fluid to the dry state with a smaller change in gas temperature and the troublesome sticky zone would be reduced to a smaller area.

We would urge all published reports on this subject to include all three points in the laboratory determination of ash fusion, and we might also point out that a very fertile field for coal research work lies in reducing the spread between softening and fluid temperatures.

MR. R. L. ROWAN.[8]—The interval between the initial deformation and the final fluid temperature might well bear further investigation. Many coals that have the same fusion temperature as determined by the A.S.T.M. method do not perform exactly the same under the same installation. I recall two coals, one from the No. 3 Pocahontas Seam and the other from Sewell Seam of the New River field. Both of these coals had about the same fusion temperature but they produced an entirely different type of clinker. One made a very objectionable clinker to handle and the other one made a fairly easy clinker to remove in the dump type multiple retort underfeed stoker. The predominant difference was in the interval between the initial deformation and the softening temperature, and the softening temperature and the fluid temperature. In the case of the coal that made the objectionable clinker the interval was very small and in the case of the coal that did not make objectionable clinker the interval was large. While I have no engineering data to support this belief my impression from seeing coals burn in a number of different installations is that possibly some more light could be thrown on this subject if more data were published and more thought was given to the interval between the initial and fluid, as well as the softening temperature.

THE CHAIRMAN (*Mr. A. C. Fieldner*[9]).—Experiments have been made on the relation between ash softening temperature and clinker formation of coals in comparison to high- and low-temperature cokes made from the same coals. Possibly Mr. Gilmore of the Canadian Department of Mines has some information on this point.

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[7] Engineering Design, The Babcock & Wilcox Co., New York City.

[8] Fuel Engineer, General Coal Co., Philadelphia, Pa.

[9] Chief, Technologic Branch, U. S. Bureau of Mines, Washington, D. C.

MR. R. E. GILMORE.[10]—Our information on the point just mentioned is limited. From a coal having a particularly low ash fusion temperature averaging about 2000 F., the high-temperature by-product coke produced had noticeable clinkers, whereas the low-temperature coke made by the Illingworth process from this particular coal showed practically no clinker trouble. The details of these tests are available in one of our publications. We attribute this difference to the lower fuel bed temperature; the low-temperature coke is considerably more reactive or, I should say, the rate of burning is higher, so that the net result is an ash bed temperature below the fusion temperature of the ash—hence no clinker trouble.

Although the reactivity of the two cokes was not determined directly we have data on the rate of burning. I believe experts give different definitions for reactivity and rate of burning.

We find that the melting range, especially for the low ash fusion coals, is a significant factor. A melting range as high as 250 as against 50 deg. Fahr. produces quite a difference in the burning of a coal; that is, assuming the same softening point of the ash. In a series of burning tests conducted in a domestic hot water boiler at the Fuel Research Laboratories on domestic coals varying widely in rank, the ash fusion temperature has been correlated with the clinkering, caking and other characteristics much along the line followed by the U. S. Bureau of Mines at Pittsburgh.

MR. G. G. RITCHIE.[11]—It is very evident that the ash softening temperature cannot be used as a sole criterion for determining the difficulty that will be encountered in burning a given coal in a particular plant or for judging the performance of one coal *versus* another. In addition to the ash content, caking, coking and other physical and chemical coal properties, the design of the boiler, furnace and appliances as well as the burning rate will materially influence the performance to be obtained from a given coal. The boiler room personnel and the human factor play an important part in the selection of a satisfactory fuel.

Practically all coals possess certain characteristics which make them suitable for use under specific conditions. Therefore, the conditions in each instance must first be ascertained before the question of the effect of ash softening temperature or any other property on performance in a given plant can be determined.

Mr. Rowan mentioned using New River in place of Pocahontas coal and stated that in a number of instances he procured better results from one coal than another where the ash softening temperature was approximately the same. I have followed up the burning of some of these New River coals averaging in ash-softening temperature from 2500 F. to 2600 F. in plants that previously specified coals having much higher fusing temperature ash and they produced very satisfactory results. To illustrate the part the human factor plays, I know of one plant in which the firing room personnel claimed they had to use a coal having high fusing temperature ash and this resulted in the purchasing agent specifying a coal with an ash softening temperature of 2800 F., containing not over 10 per cent ash. The owners of this plant were persuaded to test another coal that contained approximately 6 per cent ash and 2600 F. fusion temperature. I do not recall the exact savings, but the equivalent evaporation per pound of coal was reduced from

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[10] Senior Engineer, Fuel Research Laboratories, Division of Fuels, Canada Bureau of Mines, Ottawa, Ont., Canada.

[11] Fuel Service Engineer, Chesapeake & Ohio Railway Co., Richmond, Va.

18 to 20 per cent. I think most of this saving was made possible through overcoming certain adverse human factors.

MR. R. A. SHERMAN.[12]—Mr. Gauger has included in his paper the suggestion I gave him, namely, that one of the principal reasons why the clinkering tendencies did not always check with the indications of the ash softening temperature was that the cone was made of finely-ground and intimately-mixed ash representative of the entire ash in the coal. In the furnace, however, the ash is never intimately mixed together.

Mr. Gauger mentions this but states that he does not agree with it. I merely want to say that I still believe that this is a very important point. Let anyone take a look at pieces of ash and clinker discharged from even an underfeed stoker where the ash has the greatest opportunity to mix of any type of combustion. He will find that the clinker is still a heterogeneous mass with white pieces of unfused ash, black glassy slag, and intermediate shades of brown in various stages of fusion.

I recently went over the analyses of a number of samples of slag and ash taken in connection with the investigation of refractory service conditions in boiler furnaces made a number of years ago when I was with the Bureau of Mines. I noted that although the composition of the sample which was representative of the total ash in the ash pit was quite similar to that of the ash of the coal fired, yet the composition of pieces of slag from various parts of the furnace differed materially from the original and had, where it was determined, quite different softening temperatures.

MR. A. W. GAUGER[13] (*author's closure, by letter*).—This paper was written for the purpose of developing information which would assist Committee D-5 in preparing a pamphlet on the significance of tests of coal and coke. Such information could only result from a full and complete discussion of the subject; it could not be developed solely by the author. I am, therefore, grateful for the many thoughtful comments and the added data that have been presented in the discussion.

Most of the comments bear out the author's view that the ash softening temperature may not be considered apart from many other factors involved in coal utilization. Mr. Barkley, for example, has listed a number of such factors for a simple hypothetical case, and I am perfectly willing to admit that "with other factors suitably adjusted," a status may be reached "where the ash softening temperature has considerable significance." It is apparent that the interpretation of this temperature is not simple and general but requires experience and judgment for proper conclusions.

Mr. Nicholls raises a question which seems to hinge on the definition of the word "significance." Without any desire to quibble over this point, which, after all, is relatively unimportant, the author wishes to state his interpretation of the word in this paper as "meaning" or "import" and further to restrict it by defining "signify" as "to show by a sign." The ash softening temperature is a sign. What does it show us? With such a definition are we not in substantial agreement when I say that the "importance has been overemphasized" and Mr. Nicholls says, "we have attempted to give them a significance beyond that warranted"?

The ash softening temperature is not a fundamental property of ash. The test is entirely empirical and can only be

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[12] Fuel Engineer, Battelle Memorial Inst., Columbus, Ohio.

[13] Professor of Fuel Technology, and Director of Mineral Industries Research, The Pennsylvania State College, State College, Pa.

justified on the basis of its usefulness in interpreting the behavior of coal in utilization. The evidence now before us indicates that it is useful but with certain limitations which have been brought out very clearly in the symposium.

With reference to the fluid interval and the viscosity of molten coal ash, those two factors were omitted advisedly. The importance of the viscous behavior of slags is recognized, and by viscous behavior I refer to the temperature coefficient of viscosity—that is, the rate at which viscosity changes with temperature—rather than to the absolute viscosity. Clinkers are not formed by flow alone but by a combination of flow and solidification. In the modern coal-burning equipment operating at high rating, almost any ash will be fused. Unless this fused slag congeals in large hard masses that cannot be handled by the equipment, no clinker difficulty exists. The author's personal view is that there is need for much more experimental work on the effect of mineral composition of the coal on the behavior of the ash in the fuel bed under the varying conditions met with in practice.

LABORATORY TESTS RELATING TO CAKING, PLASTIC, GAS- AND COKE-MAKING PROPERTIES OF BITUMINOUS COALS

By O. O. Malleis[1]

A number of laboratory tests have been proposed by workers in the field of bituminous coal is an attempt to determine the relative caking, plastic, gas- and coke-making properties of bituminous coal with particular reference to the manufacture of coke. None of these methods, however, has reached the state of a tentative or standard method of the A.S.T.M. and only one method, the agglutinating value of coal,[2] has been published as information.

This condition, however, does not indicate a lack of interest in or of value of the information. The complexity of what is occurring, when bituminous coals are being heated, undoubtedly has largely accounted for the apparently slow progress in developing and standardizing methods of test for determining the relative caking, plastic, gas- and coke-making properties of bituminous coals. In 1925 the Society's Committee D-5 on Coal and Coke appointed a special subcommittee on Coking Index[3] as a section of Subcommittee I on Methods of Testing. In 1927, this special subcommittee was made a regular subcommittee of Committee D-5 under the designation of Subcommittee VI on Agglutinating Value.[4] In 1936, Committee D-5 formed Subcommittee XV on Plasticity and Swelling of Coal. This subcommittee held its first meeting at Chicago on March 1, 1937.

Investigations have been conducted in a number of laboratories throughout the world on the caking and plastic properties of bituminous coal with regard to developing methods of tests which would be an aid in the study of the coking process or in selection of coals for the manufacture of coke. In America, work on caking and plastic properties includes studies at the University of Illinois **(1)**.[5] Yale University **(2)**, several laboratories in connection with plants manufacturing coke and gas **(3, 4, 19)**, and the U. S. Bureau of Mines **(5, 6, 7, 8, 9)**.

The only comprehensive study of caking and plastic properties of bituminous coals that has ever been made is the work of the U. S. Bureau of Mines in connection with a survey of the gas- and coke-making properties of American coals. This work **(7)** was started in 1927 when interest in this problem was crystallized by the Carbonization Committee of the American Gas Assn., which recommended cooperation with the U. S. Bureau of Mines in a "study of the present-day knowledge on the testing of coal for gas- and coke-making properties." The American Gas Assn. started financial and technical cooperation **(10)** with the U. S. Bureau of Mines in 1928

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[1] Manager, Inspection Division, Appalachian Coals, Inc., Cincinnati Ohio.

[2] Proposed Method of Test for Agglutinating Value of Coal, *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 457 (1934).

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part I, p. 412 (1926).

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part I, p. 492 (1927).

[5] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 414.

and continued this cooperation during 1929, 1930, and 1931. Since 1931, the work has been actively continued by the U. S. Bureau of Mines. It was recognized, at the outset that a study such as this "should be comprehensive and broadly fundamental" and that the "chemical and physical properties of the coal and the method of heat treatment should be correlated with the yields and nature of the products obtained; furthermore, the results of laboratory investigation should be correlated as far as possible with commercial results obtained in the gas and coking industries." In addition to both high- and low-temperature carbonization tests and the usual analytical determinations, this study included determination of agglutinating value and various plastic properties according to the methods that were in the most general use or showed the most promise of yielding useful information.

All the methods and apparatus used in connection with the survey of the gas-, coke- and by-product-making properties of American coals are given in U. S. Bureau of Mines *Bulletin 344*. The complete procedure for making the carbonization tests was also published in U. S. Bureau of Mines *Monograph 5*. There is also a great deal of valuable information on the methods for making carbonization tests in the *Proceedings* of the American Gas Assn. (**11, 12, 13**).

The results of the investigation of the gas-, coke- and by-product-making properties for the first 30 coals have been summarized in U. S. Bureau of Mines *Monograph 5*. Results of the tests on some of the individual coals have been published in summarized form or in more detail in the *Proceedings* of the American Gas Assn. (**14**), *Industrial and Engineering Chemistry* (**15**), International Coal Conference (1931) (**16**), and in publications of the U. S. Bureau of Mines (**17**).

In this paper, an attempt will be made to indicate some practical significance of the tests relating to caking, plastic, gas- and coke-making properties of bituminous coals. However, because of the lack of data on a large enough range of coals in each group of the A.S.T.M. Tentative Specification for Classification of Coals by Rank (D 388 – 36 T)[6] and because of the lack of correlation of available test data with plant performance, it will be very difficult to indicate the definite practical significance of some of the tests now being used.

An outline of the tests to be discussed follows:

A. Caking Properties:
  1. Coke button from volatile matter test.
  2. Agglutinating-value test.

B. Plastic Properties:
  1. Resistance to gas flow test.
  2. Swelling test.
  3. Plastometer test.
  4. Expansion during coking.

C. Gas- and Coke-making Properties:
  1. High-temperature coking.
     (*a*) Laboratory distillation test.
     (*b*) BM-AGA carbonization test.
     (*c*) Box-coking test.
  2. Low-temperature coking.

Before discussing the individual tests, it should be pointed out that direct practical significance is not always possible. The author would prefer to think of most tests more as an aid rather than as a direct practical indication. Correlation of test data with plant performance in many cases is difficult largely because it is not feasible to reproduce, in small-scale laboratory tests, all the properties of the plant coal charge. Many properties of the coal charge are, of course, due to the characteristics of the coal itself, such as proximate and ultimate analyses,

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[6] *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 812 (1936); also 1936 Book of A. S. T. M. Tentative Standards, p. 520.

calorific value, ash-softening temperature and specific gravity of the coal, and these may be considered fairly fixed. Bulk density or cubic-foot weight, however, is a most important property of any coal charge, which may be varied over a fairly wide range, particularly because of the content of added or surface moisture, range in size of coal pieces, and size-consist.

A. CAKING PROPERTIES

Many coals when heated at a uniformly increasing temperature in the absence of air, fuse and become plastic. Fieldner and Selvig (19) designate such coals as caking coals and they point out that coals possess this caking property in varying degrees. Two methods are being used for testing caking properties.

1. Coke Button from the Volatile Matter Test:

Rose (20) has pointed out that the simplest of all coking tests is doubtless the standard volatile-matter test. The resulting coke button will indicate whether the coal is non-caking, poorly caking, or strongly caking. Such indications, of course, have to be based mainly upon visual examination of the coke button. Laboratories testing a large variety of coals will often associate a certain type of coke button with certain types of coal. Low-volatile coals when heated in the standard volatile-matter test, usually expand to many times the volume of the original coal used for the test, and often the resulting coke button will completely fill the test crucible. The fairly strong-coking high-volatile coals generally fuse quite completely and form more or less spherical coke buttons, often several times the size of the original volume of the test coal, with some coke buttons from the most strongly coking high-volatile coals as large as one half of the volume of the test crucible. Weakly caking coals do not expand to any extent, and their resulting coke button will occupy about the same volume as the coal used for the test. The coke buttons from the fairly strong-coking coals are very porous and usually contain a fairly large cavity in the central portion. However, the coke buttons from the more poorly caking coals, usually are denser, since these coals do not swell to any extent upon application of heat in the volatile-matter test. Usually the center portions of the coke button from a poorly caking coal have "dropped," and there are one or more decided fracture cracks across and through the coke button. In non-caking coals, there is practically no fusing together of the coal particles, and the residue from heating is still in granular condition or will fall apart upon the application of a slight pressure.

It is probably not generally realized to just what extent the coke button from the standard volatile-matter test is used as an aid in determining the caking property of coals. A few examples will suffice. Fischer (21) and Davis and Reynolds (22) used coke buttons from volatile-matter tests in their studies of the coking constituents of coal. Rose and Sebastian (4) also used coke buttons from volatile-matter tests in their laboratory study of moderate oxidation of coking coals during storage. The coke button as an agglomerating index (23,24) was incorporated in the Tentative Specifications for Classification of Coals by Rank (D 388 - 36 T)[6] in 1936 for determining the requisite physical properties of semi-anthracite and high-volatile *C* bituminous coals. The definition of agglomerating coals is given as follows: "Coals which in the volatile matter determination produce either an agglomerate button that will support a 500-g. weight without pulverizing, or a button showing swelling or cell structure, shall be considered agglomerating from the standpoint of classification."

Turning to the burning of coals on stokers, Tate (25) reported that he used coke buttons obtained from rapidly heating 2 g. of 100-mesh coal in a covered porcelain crucible over a bunsen flame as an aid in explaining performance of widely different coals on a chain grate or overfeed stoker. He pointed out that "one can readily visualize what difficulty would be encountered in attempting to burn a highly swelling coal on a grate where there was little or no agitation." He also stated that "although not to the same extent, this information (type of coke button) is of value in determining the results to be obtained in an underfeed stoker."

TABLE I.—AGGLUTINATING VALUES FOR STRAIGHT COALS.
U. S. Bureau of Mines *Monograph 5* and *Technical Papers 570* and *571*.

Coal	State	County	Bed	Moisture as Carbonized, per cent	Dry Basis		Calorific Value, Dry, Ash-Free Basis, B.t.u. per lb.	Agglutinating Value	
					Volatile Matter, per cent	Ash, per cent		Crushing Strength, kg.	Coal-Sand Ratio
No. 23......	West Virginia	McDowell	Pocahontas No. 4	0.8	15.5	4.8	15 750	16.3	1:10
No. 26......	West Virginia	Raleigh	Sewell	1.3	21.1	2.1	15 720	9.7	1:15
								5.6	1:20
								3.3	1:25
No. 27......	West Virginia	Fayette	Sewell	1.9	27.0	2.4	15 640	7.6	1:15
								4.8	1:20
								2.9	1:25
No. 4......	Maryland	Garrett	Davis	1.4	22.4	9.9	15 560	12.2	1:10
No. 31......	Virginia	Buchanan	Clintwood	1.9	32.1	5.7	15 520	8.3	1:15
								4.4	1:20
								2.9	1:25
								2.1	1:30
No. 8......	Alabama	Jefferson	Mary Lee W.	4.2	28.8	8.7	15 490	13.0	1:10
No. 18......	Alabama	Jefferson	Pratt W.	3.0	32.5	3.2	15 450	9.9	1:10
No. 3......	Virginia	Wise	Taggart	1.7	36.7	2.7	15 450	5.7	1:10
No. 11......	Canada	British Columbia	B	1.4	27.4	6.3	15 410	14.6	1:10
No. 17......	Alabama	Jefferson	Pratt	1.5	30.6	9.3	15 380	12.7	1:10
No. 9......	Pennsylvania	Fayette	Pittsburgh	1.9	34.3	7.6	15 350	10.9	1:10
No. 12......	Pennsylvania	Fayette	Pittsburgh	1.0	33.9	8.6	15 270	9.7	1:10
No. 28......	Pennsylvania	Allegheny	Pittsburgh	1.8	35.7	5.6	15 240	7.6	1:15
								5.3	1:20
								3.3	1:25
No. 16......	West Virginia	Boone	Alma	2.1	38.5	7.3	15 210	9.3	1:10
No. 22......	West Virginia	Marion	Pittsburgh	1.9	38.0	6.1	15 210	13.3	1:10
No. 7......	Alabama	Jefferson	Mary Lee	1.2	28.2	16.1	15 200	11.3	1:10
No. 1......	Pennsylvania	Allegheny	Pittsburgh	1.6	34.8	7.6	15 190	6.7	1:10
No. 14......	West Virginia	Logan	Chilton	2.2	37.0	4.4	15 190	8.4	1:10
No. 1W.....	Pennsylvania	Allegheny	Pittsburgh W.	2.4	35.3	6.0	15 170	9.7	1:10
No. 20......	Pennsylvania	Allegheny	Thick Freeport	2.2	37.1	6.4	15 160	10.2	1:10
No. 15......	West Virginia	Kanawha	No. 2 Gas	1.9	39.6	2.8	15 150	7.1	1:10
No. 2......	Kentucky	Letcher	Elkhorn	2.2	37.4	2.2	15 070	4.3	1:10
No. 13......	Alabama	Walker	Black Creek	3.1	37.2	2.7	15 050	6.7	1:10
No. 32......	Pennsylvania	Washington	Pittsburgh	2.0	42.1	8.2	14 810	7.8	1:15
								5.1	1:20
								3.2	1:25
								2.3	1:30
No. 19......	Utah	Carbon	L. Sunnyside	4.6	40.7	6.3	14 570	3.9	1:10
No. 10......	Illinois	Franklin	No. 6	7.9	34.9	13.4	14 460	4.3	1:10
No. 21......	Kentucky	Muhlenberg	Green River	10.1	40.3	7.5	14 400	8.6	1:10

NOTE.—Coals Nos. 1 to 23, inclusive: Marshall-Bird Agglutinating Index.
Coals Nos. 24 to 32, inclusive: U. S. Bureau of Mines Method.

The examination of the coke button from the standard volatile-matter test has long been used as the first step in an estimation of the caking properties of coals. Undoubtedly there are many cases where the type of these coke buttons would have value if correlations were made for definite kinds of industrial application of coals such as reported by Tate in connection with a chain grate. There is a real need for information with regard to the probable caking tendencies when coals are burned on stokers, both overfeed and underfeed, and both industrial and residential. Too much, however, should not be expected of the examination of these coke buttons. They should be viewed with regard to serving as an aid. Of course, the wider the range in the caking characteristics, the more information such an examination should yield. The test is so simple and so available that all possible applications should be completely explored.

TABLE II.—AGGLUTINATING VALUES FOR MIXED COALS.
U. S. Bureau of Mines *Monograph 5.*

Coal	Kind of Coal	Moisture as Carbonized, per cent	Dry Basis		Calorific Value, Dry, Ash-Free Basis, B.t.u. per lb.	Agglutinating Value	
			Volatile Matter, per cent	Ash, per cent		Crushing Strength, kg.	Coal-Sand Ratio
No. 18A	80% Coal No. 17, Pratt; 20% Coal No. 7, Mary Lee	3.4	29.2	6.4	15 520	10.1	1:1
No. 17	100% Pratt	1.5	30.6	9.3	15 380	12.7	1:10
No. 7	100% Mary Lee	1.2	28.2	16.1	15 200	11.3	1:10
No. 5	40% Coal No. 1 W. Pittsburgh; 60% Coal No. 4, Davis	1.5	28.1	7.6	15 460	11.6	1:10
No. 6	80% Coal No. 1 W. Pittsburgh; 20% Coal No. 4, Davis	1.3	33.7	5.5	15 240	10.6	1:10
No. 1W	100% Pittsburgh W.	2.4	35.3	6.0	15 170	9.7	1:10
No. 4	100% Davis	1.4	22.4	9.9	15 560	12.2	1:10
No. 30	80% Coal No. 28, Pittsburgh; 20% Coal No. 27, Sewell	1.9	34.3	4.8	15 340	7.8	1:15
No. 28	100% Pittsburgh	1.8	35.7	5.6	15 240	7.6	1:15
No. 27	100% Sewell	1.9	27.0	2.4	15 640	7.6	1:15
No. 29	80% Coal No. 22, Pittsburgh; 20% Coal No. 26, Sewell	1.8	32.6	4.9	15 330	8.2	1:15
No. 22	100% Pittsburgh	1.9	38.0	6.1	15 210	13.3	1:10
No. 26	100% Sewell	1.3	21.1	2.1	15 720	9.7	1:15
No. 24	80% Coal No. 22, Pittsburgh; 20% Coal No. 23, Pocahontas No. 4	1.3	32.9	6.0	15 270	10.2	1:10
No. 22	100% Pittsburgh	1.9	38.0	6.1	15 210	13.3	1:10
No. 23	100% Pocahontas No. 4	0.8	15.5	4.8	15 750	16.3	1:10

NOTE.—Coals 1 to 23 inclusive: Marshall-Bird Agglutinating Index.
Coals 24 to 30 inclusive: U. S. Bureau of Mines Method.

2. Agglutinating-Value Test:

Agglutinating values for coal in the proposed method[1] are obtained by crushing buttons made by carbonizing mixtures of 60 to 45-mesh Ottawa sand and 0 to 200-mesh coal in a standard volatile-matter furnace under prescribed conditions. The tests are made at the following ratios of sand to coal: 15:1; 20:1; 25:1; and 30:1. Marshall and Bird (5) in their earlier investigation, used 10 parts sand and 1 part coal.

The scope of the proposed method[1] states that: "This method is a small-scale laboratory test for obtaining information regarding the coking and caking properties of coal. It is an approximate measure of that material in coal which fuses and becomes plastic on heating."

The term "coking property" is a very general term and rather hard to define. Rose and Sebastian (4) point out that "most existing definitions do not describe the property, but refer instead to the quality of the coke which can be obtained." In general, strongly caking or coking means: (1) on industrial stokers, a coking or matting over of the fuel bed which cannot be readily broken up by the normal operation of the stoker; (2) on the residential stokers, the formation of coke trees which materially disturb the fuel bed; and (3) on the coke plant, the production of a coke of a desired size which will stand the necessary mechanical handling without the production of excessive breeze coke.

Considerable data on agglutinating values are available in the reports of the U. S. Bureau of Mines on their investigation of the gas- and coke-making properties of American coals. Table I contains data for straight coals, and Table II for mixed coals. The coals have been arranged according to their dry, ash-free calorific values.

An examination of the agglutinating values in Table I fails to reveal any definite trend or relationship. In Table II the usual improving effect of addition of low-volatile to a high-volatile coal when such mixtures are coked, is not always indicated. Fieldner and Davis (**10**), commenting on the agglutinating values, say: "Individual coals of approximately the same index vary widely in the stability of the coke produced. These deviations are of sufficient magnitude to invalidate the numerical agglutinating index at a single ratio as a reliable indicator of the strength or stability of the coke that can be produced from the coal in question."

The U. S. Bureau of Mines (**27**) has compared the agglutinating values of a number of low-volatile coals with operating observations for burning coal on stokers. The conclusions were (**19**) that "in general there is a fair correlation between results of the agglutinating-value test and the coking tendencies and the nature of coke formed on burning various coals in natural-draft equipment. For forced-draft equipment, the correlation was more erratic. More work needs to be done along these lines to show the practical application of this test."

Agglutinating indices do seem to have value for special purposes. Rose and Sebastian (**4**) used the agglutinating test in their study of the changes in properties of coking coals due to moderate oxidation during storage. They point out that "in the case of the coals which they tested (Pocahontas No. 3, Elkhorn No. 3 and Powellton seams), the agglutinating values were enormously more affected by oxidation than were proximate and ultimate analyses and calorific values." It would seem that the agglutinating-value test may have value as an indicator of oxidation and weathering. Fieldner and Davis (**10**) say that "a series of increasing coal-sand ratios, may prove to be a useful auxiliary test for special purposes."

Summarizing, two general statements probably can safely be made, based on the reported results of investigations of the agglutinating-value test:

1. The agglutinating-value test has been found to have value for special investigation such as detecting deterioration of coking properties of coal due to storage.
2. The agglutinating-value test seems to have little value as a reliable index of the probable caking or coking properties of a coal.

B. PLASTIC PROPERTIES

The tests to be considered in this discussion of plastic properties of coal are those which have been most largely used and for which there are the most related data. As in the agglutinating-value test, the reports of the U. S. Bureau of Mines of their investigation of the gas- and coke-making properties of American coals are again the source of our test data. Two tables of data, according to the three tests used by the U. S. Bureau of Mines, have been included to show the nature and trend of the results which have been reported to date. Table III contains data on plastic properties for straight coals, and Table IV contains data for mixed coals.

TABLE III.—PLASTIC RANGE DATA FOR STRAIGHT COALS.

Tests by U. S. Bureau of Mines[a, b]

Coal	State	County	Bed	Moisture as Carbonized, per cent	Dry Basis		Calorific Value, Dry, Ash-Free Basis, B.t.u. per lb.	Modified Layng-Hathorne Test[c]			Agde-Damm Test[d]			Davis Plastometer Test[e]					
								Plastic Range, deg. Cent.							Maximum Fluidity		Maximum Resistance		
					Volatile Matter, per cent	Ash, per cent		Pressure Developed	Pressure Dropped	Maximum Pressure, mm. water	Initial Contraction, Temperature, deg. Cent.	Initial Expansion, Temperature, deg. Cent.	Swelling Coefficient	Resistance Develops, deg. Cent.	in-lb.	deg. Cent.	in-lb.	deg. Cent.	Resistance Ends, deg. Cent.
No. 41......	West Virginia	McDowell	Beckley	2.3	18.3	3.9	15 780	445	525	190	410	454	1.0	447	1.8	469	72.0	503	515
No. 23[f].....	West Virginia	McDowell	Pocahontas No. 4	......	....	....	......	.....	......	.....	439	above 500	1.0	Slight intermittent resistance					
No. 23......	West Virginia	McDowell	Pocahontas No. 4	0.8	15.5	4.8	15 750	448	547	348	407	462	1.1	465	1.2	469	3.6	468	474
No. 26......	West Virginia	Raleigh	Sewell	1.3	21.1	2.1	15 720	415	534	600	378	431	1.4	414	8.4	447	43.9	436	469
No. 27......	West Virginia	Fayette	Sewell	1.9	27.0	2.4	15 640	393	526	910	320	396	2.2	415	2.9	431	29.7	421	465
No. 4......	Maryland	Garrett	Davis	1.4	22.4	9.9	15 560	412	498	774	355	408	1.5	411	8.0	423	25.8	415	427
No. 31......	Virginia	Buchanan	Clintwood	1.9	32.1	5.7	15 520	380	475	435	301	384	3.2	386	2.4	428	21.4	407	451
No. 8......	Alabama	Jefferson	Mary Lee W.	4.2	28.8	8.7	15 490	384	461	520	319	380	3.6	393	6.0	434	49.0	393	457
No. 18......	Alabama	Jefferson	Pratt W.	3.0	32.5	3.2	15 450	404	458	233	301	384	1.8	405	2.5	428	17.7	419	451
No. 3......	Virginia	Wise	Taggart	1.7	36.7	2.7	15 450	420	453	380	314	387	1.7	410	3.1	431	17.4	413	448
No. 11......	Canada	British Columbia	B	1.4	27.4	6.3	15 410	406	507	400	335	409	1.4	426	12.1	434	47.7	431	453
No. 36......	West Virginia	Mingo	Alma	2.4	36.6	3.3	15 390	384	431	195	312	409	1.7	403	0.2	434	26.0	464	490
No. 17......	Alabama	Jefferson	Pratt	1.5	30.6	9.3	15 380	407	464	218	307	394	1.6	398	5.0	434	18.6	418	456
No. 9......	Pennsylvania	Fayette	Pittsburgh	1.9	34.3	7.6	15 350	394	455	250	300	375	3.0	393	3.5	425	25.3	406	451
No. 40......	West Virginia	Mingo	Lower Cedar Grove	1.8	35.9	4.4	15 340	414	447	199	334	413	1.3	415	0.5	440	41.0	467	478
No. 12......	Pennsylvania	Fayette	Pittsburgh	1.0	33.9	8.6	15 270	374	454	280	296	378	2.5	391	4.4	429	32.3	401	459
No. 28......	Pennsylvania	Alleghany	Pittsburgh	1.8	35.7	5.6	15 240	382	443	340	298	378	2.4	393	4.6	421	48.5	404	447
No. 39......	West Virginia	Mingo	Upper Cedar Grove	1.8	36.9	6.9	15 230	395	444	88	308	405	1.2	406	0.8	440	40.0	479	490
No. 16......	West Virginia	Boone	Alma	2.1	38.5	7.3	15 210	383	438	177	267	371	2.1	396	0.8	440	5.1	435	447
No. 22......	West Virginia	Marion	Pittsburgh	1.9	38.0	6.1	15 210	387	439	180	286	376	2.3	390	4.9	419	34.4	400	442
No. 7......	Alabama	Jefferson	Mary Lee	1.2	28.2	16.1	15 200	382	450	371	322	386	1.6	393	3.0	423	43.0	398	446
No. 1......	Pennsylvania	Allegheny	Pittsburgh	1.6	34.8	7.6	15 190	402	454	523	320	390	1.7	....	....	....	....	....	....
No. 14......	West Virginia	Logan	Chilton	2.2	37.0	4.4	15 190	385	433	188	304	388	1.5	395	0.5	417	13.2	408	431
No. 1W....	Pennsylvania	Allegheny	Pittsburgh W.	2.4	35.3	6.0	15 170	395	447	263	291	372	2.4	387	7.8	421	34.7	397	443
No. 20......	Pennsylvania	Allegheny	Thick Freeport	2.2	37.1	6.4	15 160	381	436	178	293	380	1.6	393	0.9	440	24.5	409	444
No. 15......	West Virginia	Kanawha	No. 2 Gas	1.9	39.6	2.8	15 150	388	472	260	287	379	1.1	406	0.0	429	6.1	415	440
No. 2......	Kentucky	Letcher	Elkhorn	2.2	37.4	2.2	15 070	426	490	255	340	394	1.2	423	5.4	437	17.0	429	443
No. 13......	Alabama	Walker	Black Creek	3.1	37.2	2.7	15 050	388	475	172	312	392	1.1	408	3.8	435	9.4	420	441
No. 33......	Kentucky	Johnson	Millers Creek	3.3	39.1	2.8	14 820	None		37	360	above 490	1.0	No resistance					
No. 32......	Pennsylvania	Washington	Pittsburgh	2.0	42.1	8.2	14 810	356	427	305	271	363	2.9	378	1.6	398	32.0	381	436
No. 19......	Utah	Carbon	L. Sunnyside	4.6	40.7	6.3	14 570	390	443	53	295	394	1.0	No resistance					
No. 10......	Illinois	Franklin	No. 6	7.9	34.9	13.4	14 460	405	450	26	275	414	1.0	No resistance					
No. 21......	Kentucky	Muhlenberg	Green River	10.1	40.3	7.5	14 400	401	450	55	236	398	0.9	No resistance					

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[a] *Monograph 5*, U. S. Bureau of Mines; *Technical Papers 570 and 571; Industrial and Engineering Chemistry*, Analytical Edition, Vol. 8, p. 443 (1936).
[b] Proximate analyses and calorific values for Coals Nos. 33 to 41, inclusive, obtained by letter.
[c] Layng-Hathorne Test.—Starting with Coal No. 33, 0- to 20-mesh coal used in place of 40- to 20-mesh coal.
[d] Agde-Damm Test.—Starting with Coal No. 33, 0- to 60-mesh coal used in place of 0- to 100-mesh coal.
[e] Davis Plastometer.—Starting with Coal No. 33, 0- to 20-mesh coal used in place of 40- to 20-mesh coal.
[f] Test according to new conditions; see footnotes [c], [d] and [e].

TABLE IV.—PLASTIC RANGE DATA FOR MIXED COALS.

[d] Agde-Damm Test.—Starting with Coal No. 33, 0- to 60-mesh coal used in place of 0- to 100-mesh coal.
[e] Davis Plastometer.—Starting with Coal No. 33, 0- to 20-mesh coal used in place of 40- to 20-mesh coal.
[f] Test according to new conditions; see footnotes [c], [d] and [e].

TABLE IV.—PLASTIC RANGE DATA FOR MIXED COALS.
Tests by U. S. Bureau of Mines[a, b].

Coal	Kind of coal	Moisture as Carbonized, per cent	Dry Basis		Calorific Value, Dry, Ash-Free Basis, B.t.u. per lb.	Modified Layng-Hathorne Test[c]			Agde-Damm Test[d]			Davis Plastometer Test[e]					
						Plastic Range, deg. Cent.							Maximum Fluidity		Maximum Resistance		
			Volatile Matter, per cent	Ash, per cent		Pressure Developed	Pressure Dropped	Maximum Pressure, mm. water	Initial Contraction, Temperature, deg. Cent.	Initial Expansion, Temperature, deg. Cent.	Swelling Coefficient	Resistance Develops, deg. Cent.	in-lb.	deg. Cent.	in-lb.	deg. Cent.	Resistance Ends, deg. Cent.
No. 18A	80% of Coal No. 17, Pratt; 20% Coal No. 7, Mary Lee	3.4	29.2	6.4	15 520	380	472	373	317	392	2.5	385	8.5	431	45.0	416	463
No. 17	100% Pratt	1.5	30.6	9.3	15 380	407	464	218	307	394	1.6	398	5.0	434	18.6	418	456
No. 7	100% Mary Lee	1.2	28.2	16.1	15 200	382	450	371	322	386	1.6	393	3.0	423	43.0	398	446
No. 5	40% Coal No. 1 W, Pittsburgh; 60% Coal No. 4, Davis	1.5	28.1	7.6	15 460	407	487	307	321	390	1.6	388	7.5	425	26.3	418	456
No. 6	80% Coal No. 1 W, Pittsburgh; 20% Coal No. 4, Davis	1.3	33.7	5.5	15 240	399	466	414	319	382	1.5	407	13.8	427	35.5	412	449
No. 1W	100% Pittsburgh	2.4	35.3	6.0	15 170	395	447	263	291	372	2.4	387	7.8	421	34.7	397	443
No. 4	100% Davis	1.4	22.4	9.9	15 560	412	498	774	355	408	1.5	411	8.0	423	25.8	415	427
No. 30	80% Coal No. 28, Pittsburgh; 20% Coal No. 27, Sewell	1.9	34.3	4.8	15 340	389	442	280	302	386	1.7	394	7.9	430	43.3	411	451
No. 28	100% Pittsburgh	1.8	35.7	5.6	15 240	382	443	340	298	378	2.4	393	4.6	421	48.5	404	447
No. 27	100% Sewell	1.9	27.0	2.4	15 640	393	526	910	320	396	2.2	415	2.9	431	29.7	421	465
No. 29	80% Coal No. 22, Pittsburgh; 20% Coal No. 26, Sewell	1.8	32.6	4.9	15 330	385	444	255	290	377	2.7	389	9.3	424	30.7	411	448
No. 22	100% Pittsburgh	1.9	38.0	6.1	15 210	387	439	180	286	376	2.3	390	4.9	419	34.4	400	442
No. 26	100% Sewell	1.3	21.1	2.1	15 720	415	534	600	378	431	1.4	414	8.4	447	43.9	436	469
No. 24	80% Coal No. 22, Pittsburgh; 20% Coal No. 23, Pocahontas No. 4	1.3	32.9	6.0	15 270	383	472	367	297	390	1.4	401	22.7	422	23.9	423	442
No. 22	100% Pittsburgh	1.9	38.0	6.1	15 210	387	439	180	286	376	2.3	390	4.9	419	34.4	400	442
No. 23	100% Pocahontas No. 4	0.8	15.5	4.8	15 750	448	547	348	407	462	1.1	465	1.2	469	3.6	468	474
No. 34	70% Coal No. 33, Millers Creek; 30% Coal No. 23, Pocahontas No. 4	3.3	32.1	3.5	14 980	None		31	372	above 500	1.0	Slight Intermittent Resistance					
No. 35	30% Coal No. 33, Millers Creek; 70% Coal No. 28 Pittsburgh	2.3	36.5	4.8	15 090	391	435	122	326	409	1.0	402	1.4	431	18.4	470	475
No. 33	100% Millers Creek	3.3	39.1	2.8	14 820	None		37	360	above 490	1.0	No Resistance					
No. 23[f]	100% Pocahontas No. 4	0.8	15.5	4.8	15 750	....	....	....	439	above 500	1.0	Slight Intermittent Resistance					
No. 28[f]	100% Pittsburgh	1.8	35.7	5.6	15 240	....	....	....	315	391	2.4	....	....	....	....	....	...
No. 37	80% Coal No. 36, Alma; 20% Coal No. 23, Pocahontas No. 4	1.4	32.4	3.7	15 420	385	432	143	335	419	1.0	416	1.0	437	26.0	467	490
No. 38	70% Coal No. 36, Alma; 30% Coal No. 23, Pocahontas No. 4	1.5	30.7	3.8	15 430	379	434	169	346	425	1.0	421	0.4	435	34.0	485	494
No. 36	100% Alma	2.4	36.6	3.3	15 390	384	431	195	312	409	1.7	403	0.2	434	26.0	464	490
No. 23[f]	100% Pocahontas No. 4	0.8	15.5	4.8	15 750	....	....	....	439	above 500	1.0	Slight Intermittent Resistance					
No. 39A	80% Coal No. 39, U. Cedar Grove; 20% Coal No. 41, Beckley	1.6	33.1	6.3	15 320	384	448	106	331	418	1.0	417	0.8	439	82.0	483	489
No. 39B	70% Coal No. 39, U. Cedar Grove; 30% Coal No. 41, Beckley	1.4	31.2	5.7	15 380	383	447	116	340	418	1.1	420	1.4	440	82.0	485	491
No. 39	100% Upper Cedar Grove	1.8	36.9	6.9	15 230	395	444	88	308	405	1.2	406	0.8	440	40.0	479	490
No. 41	100% Beckley	2.3	18.3	3.9	15 780	445	525	190	410	454	1.0	447	1.8	469	72.0	503	515
No. 40A	80% Coal No. 40, L. Cedar Grove; 20% Coal No. 41, Beckley	1.9	31.9	4.1	15 410	404	451	178	338	413	1.3	418	1.0	443	58.0	472	487
No. 40B	70% Coal No. 40, L. Cedar Grove; 30% Coal No. 41, Beckley	2.0	30.4	4.1	15 450	400	....	145	344	414	1.3	414	2.0	443	75.0	480	487
No. 40	100% Lower Cedar Grove	1.8	35.9	4.4	15 340	414	447	199	334	413	1.3	415	0.5	440	41.0	467	478
No. 41	100% Beckley	2.3	18.3	3.9	15 780	445	525	190	410	454	1.0	447	1.8	469	72.0	503	515

[a] *Monograph 5*, U. S. Bureau of Mines; *Technical Papers 570 and 571*; *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 8, p. 443 (1936).
[b] Proximate analyses and calorific values for Coals Nos. 33 to 41, inclusive, obtained by letter.
[c] Layng-Hathorne Test.—Starting with Coal No. 33, 0- to 20-mesh coal used in place of 40- to 20-mesh coal.
[d] Agde-Damm Test.—Starting with Coal No. 33, 0- to 60-mesh coal used in place of 0- to 100-mesh coal.
[e] Davis Plastometer.—Starting with Coal No. 33, 0- to 20-mesh coal used in place of 40- to 20-mesh coal.
[f] Test according to new conditions; see footnotes [c], [d] and [e].

1. Resistance to Gas Flow Test:

The method used by the U. S. Bureau of Mines to determine the resistance to gas flow is a modification of that described by Layng and Hathorne (1, 7). In this test, nitrogen is passed through a short column of a mixture[7] of 10 parts of coal and 4 parts of electrode graphite while being heated in a glass tube held in a vertical position at prescribed conditions. The temperatures are noted when resistance develops in the passage of gas and when the resistance drops.

Usually this method has been referred to as a melting-point method, and the temperature where resistance develops is considered the melting point of the coal under the conditions of this test.

For the 32 coals listed in Table III, the low-volatile coals have the highest melting point, approximately 445 C. The melting points of most of the high-volatile coals were in the range of 380 to 400 C. The low- and medium-volatile coals had decidedly longer plastic ranges, from 80 to 133 C. The plastic range for most of the high-volatile coals was from about 45 to 60 C. Only one coal in this group, a high-volatile coal from the Millers Creek seam, showed no plastic range. These data seem the most significant to stress for the straight coals. In Table IV, it is of interest to note that the melting point of the mixed coal is often slightly lower than that of either coal used in the mixture.

Brewer and Atkinson (9) say: "From a study of a large number of Layng-Hathorne curves, the authors believe that the test is not capable of giving any reliable information which is not given to a fuller extent by the other tests." (Agde-Damm and Davis plastometer.)

With regard to practical significance, it is the understanding of the author that the Layng-Hathorne test has been used by a large by-product coke plant to group together high-volatile coals of similar melting point in order to produce better coke from a physical standpoint when coking straight high-volatile coal.

2. Swelling Test:

The Agde-Damm test was selected by the U. S. Bureau of Mines to measure linear expansion or contraction of a test sample. This is accomplished by measuring the linear expansion and contraction which a small cylindrical briquet of coal undergoes when being heated to 500 C. In this test the temperatures are noted when the coal briquet first contracts and when it first expands. In this test, another briquet is allowed to expand freely without the weight of an indicating mechanism resting upon it and the ratio of the apparent volume of the coke to the apparent volume of the coal is called the "swelling coefficient" or "free expansion swelling coefficient."

It will be noted that the temperature of initial contraction is materially lower for all coals in comparison with the corresponding temperatures when pressure or resistance develops in the Layng-Hathorne and plastometer tests, respectively. There is fairly close agreement between the temperature of initial expansion in the Agde-Damm with the temperature where resistance develops in the plastometer test.

With regard to the practical significance of the data from the Agde-Damm, comparisons with plant performance will have to be made to show the practical application of this test.

3. Plastometer Test:

The plastometer test was developed by J. D. Davis of the U. S. Bureau of

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[7] For coals starting with No. 33 in Tables III and IV the electrode graphite was eliminated and the length of the column of coal was reduced about two thirds (9).

Mines. This test method makes a "continuous measurement of the viscosity of the coal while it is being heated through the plastic state."

In this test, coal is stirred in a retort while being heated. "When the temperature reaches the plastic range, the coal sample becomes viscous, and resistance to rotation develops rapidly. With higher temperatures the resistance usually decreases because the coal becomes more fluid. As the heating is continued further, chemical changes in the coal cause it to become more and more viscous and finally to solidify. When solidification occurs, the coke formed is quickly broken up and resistance to rotation practically ceases, this indicating the end of the plastic range." The best type of report of these data is a plot for each test, but individual plots are hard to compare. When numerical values are reported, the following values are shown: temperatures when resistance develops and ends; temperatures at maximum fluidity and resistance; and the resistance to rotation at maximum fluidity and maximum resistance.

In studying the plastometer data in Tables III and IV, the need for data for the lower volatile coals is immediately evident. However, judging from the coals represented, the total plastic temperature range appears to be of value in trying to make practical application of the results of this test. The following points seem significant:

1. Comparing the plastic ranges for the plastometer and Layng-Hathorne tests:

(*a*) Low- and medium-volatile coals do not have nearly so wide a plastic range for the plastometer test as for the Layng-Hathorne test.

(*b*) High-volatile coals having a calorific value of more than 15,000 B.t.u. per lb. on the dry, ash-free basis have fairly similar plastic ranges in both tests, except that the range is narrower for some of the high-volatile coals in case of the plastometer test.

(*c*) Four out of five high-volatile coals, with dry, ash-free calorific values of less than 15,000 B.t.u. per lb., showed no plastic range in the plastometer test as compared with only one out of those same five coals in the Layng-Hathorne test.

2. The plastic range values for the plastometer test do not seem to have any definite relationship to the percentage of volatile matter.

3. The range during which high-volatile coals are plastic varies from 20 to 87 C. for the coals in Tables III and IV which showed resistance in the plastometer test.

4. Many high-volatile coals, having a calorific value of less than approximately 15,000 B.t.u. per lb. on the dry, ash-free basis, probably will offer little or no resistance, that is, have no plastic range in the plastometer test. There was one exception in five coals reported.

With regard to practical application of plastometer data in connection with the manufacture of coke, probably the following comments would be of interest:

1. Judging from past experience in the coking of various coals, it would seem that the plastic range values for high-volatile coals may be indicative, in a general way, of the physical quality of the resultant coke which would be produced if the high-volatile coal in question were coked straight. As nearly as the author can recall, the high-volatile coals in Table III, having the lowest plastic range, would be expected to produce the poorest quality of coke from an over-all physical standpoint when coked straight in a standard by-product oven under ordinary conditions.

2. More information is needed on low-volatile coals before any comments

are justified on the value of their plasticity data. It has been the author's hope that plasticity values may aid in selecting low-volatile coals for admixture with high-volatile coals for the production of coke of the best over-all physical quality.

With regard to application of plastometer data to stokers, the data have been studied with a view to determine whether this test would indicate that a coal would, in a general way, be considered as "free-burning" or "caking" when burned on a grate, and, if possible, to what degree. Of course, it is realized that "free-burning" and "caking" as applied to stokers are very general terms and probably cannot be defined exactly. The plastic range values, however, look very promising as an aid in this kind of classification as far as high-volatile coals are concerned. Certainly the coals which offer no resistance in the Davis plastometer would generally be classed as "free-burning." As the plastic range values increase in high-volatile coals, the indication that such coals would be classed as more and more strongly coking seems to be generally in accord with what would be expected in burning on stokers. Too few low-volatile coals have been tested to make any statement about their plastic range values as far as stokers are concerned.

Summarizing with regard to the three plastic tests, the author would agree with Brewer and Atkinson (9) that "in general, the Davis plastometer gives much more complete information in the plastic range." Based on the small amount of data available, the plastometer data seem to have definite practical significance both in the carbonization and the burning of coals on stokers. More data on a larger variety of coals, particular low- and medium-volatile coals, are needed.

4. *Expansion During Coking:*

Considerable interest has developed in coal expansion as related to the by-product coke oven after the publication by Altieri (18) in 1935, of the description of an apparatus to measure expansion throughout the entire coking period. Up to this time, practically no results of work done in this country on expansion of coals as related to coke ovens have been reported, in spite of the great constant interest taken in this subject by the entire by-product coking industry.

In the Altieri test a charge of about 8 lb. of coal is heated from one direction and the force of expansion is measured by a suitable mechanism. The test coal should be of the same size and moisture content or, in other words, of the same bulk density as this coal would be when charged into a coke oven.

Active work is being done by a number of companies, but so far practically no information has been published with regard to the results being obtained. Until such test information becomes available for a number of coals, little can be said about the practical significance of this test. It is believed, however, that the test will yield valuable information, especially on borderline coals, also about the behavior of the coal charge during carbonization between the time of charging and pushing.

C. GAS- AND COKE-MAKING PROPERTIES

In the testing of coals for the manufacture of coke, nearly all the yield data and characteristics of the various products can be fairly satisfactorily determined by laboratory or small-scale tests except the physical character of the coke which may be produced in a coke oven. The methods of test which have been generally used will be discussed.

1. High-Temperature Coking:

(*a*) *Laboratory Distillation Tests.*—The procedure that has been most generally used in the United States, as well as in many other countries, for evaluation of coal by a laboratory distillation test is the procedure published by the Chemists Committee of the U. S. Steel Corp. (26). Usually this test has been designated as the "tube test." In this test, 20 g. of 35-mesh dry coal are heated progressively up to 900 C. or even to a somewhat higher temperature in a transparent quartz tube held in a horizontal position usually in a gas-fired furnace.

The tube test gives yields of coke, gas, tar, ammonia, both free and fixed, and light oil. There is also sufficient gas for analysis from which the calorific value may be calculated. By careful standardization of the test with specific commercial plants and with intelligent interpretation (10) of the results, useful information can be obtained from tube test results. In view of the small quantity of coal used and the consequent small quantities of the various products, the tube test should be considered mainly as a test for determination of yields of products from the carbonization of coal.

(*b*) *BM-AGA Carbonization Test.*—In the Bureau of Mines—American Gas Assn. carbonization tests, from 85 to 175 lb. of coal may be carbonized depending upon the diameter of the retort, which ranges from 13 to 18 in. in diameter. Carbonization tests may be made from 500 to 1100 C. and sufficient yields of all products are obtained for any desired analytical examination. The latter is a very desirable feature.

In U. S. Bureau of Mines *Monograph 5*, a number of comparisons with plant practice were made and the following relations are shown:

Most of the plant yields of coke, gas and B.t.u. in gas per pound of coal fall between the 900 and 1000 C. carbonization results.

Light oil yields agree best with the 900 C. results.

Ammonia test yields are all too low because of decomposition of ammonia by the iron retort.

Tar yields show best agreement at 1000 C. with the plant yields.

The apparent specific gravity of the test coke was approximately 0.1 less than for by-product-oven cokes.

The Bureau of Mines—American Gas Assn. carbonization test is unquestionably the best large-scale carbonizing procedure that has been developed to date. The yields at the proper carbonizing temperature are surprisingly close to plant yields. It is unfortunate that the ammonia yields are so low because of decomposition by the iron retort. Because of the lower apparent specific gravity of the test coke, the physical quality of the coke is not so satisfactory as would be desired.

(*c*) *Box-Coking Test.*—Box-coking tests are made in regular commercial coke ovens and are usually for the purpose of obtaining information about the physical character of the coke. Next to full-scale oven tests, box-coking tests, if properly made, are in general the most reliable means of determining the physical character of the resultant coke.

Summarizing with regard to tests for evaluating coals for the manufacture of high-temperature coke:

1. If only yield data and calorific value of the resultant gas are required, the laboratory distillation or tube test is the logical method to employ.

2. If examination of the various products of carbonization is also required, then a procedure like the Bureau of Mines—American Gas Assn. carbonization test seems the most logical method to employ.

3. Although the Bureau of Mines—American Gas Assn. test yields considerable valuable information about the physical quality of the coke, box-coking tests properly made in a commercial coke oven should be included in a testing program in order to evaluate the physical character of the coke which may be produced. If the expense can be justified, final confirmation of the physical character of the coke should be made by full-oven tests.

2. *Low-Temperature Coking:*

The Fischer low-temperature assay is the laboratory procedure very largely used for evaluation of coals for low-temperature coking. In this test, 50 g. of coal are distilled at a temperature of 500 C.

The following comments are from U. S. Bureau of Mines *Monograph 5*: "The tar yield obtained in commercial low-temperature plants usually is about 70 per cent of the Fischer assay yields because of decomposition of part of the primary tar in the retort. Decomposition increases with carbonizing temperature, so that in high-temperature carbonization the tar yield may be only one third that obtainable in the low-temperature assay. The assay is valuable, however, because it indicates the maximum amount of this by-product obtainable from a given coal when heated at a low temperature and the tar removed quickly with minimum opportunity for secondary decomposition; it therefore indicates the characteristics of the coal rather than results to be expected from the retort or coke oven. For this purpose the test may be of value to practical engineers in the gas or coking industry. It is useful for indicating the theoretical yields of liquid by-products on low-temperature distillation of coal."

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DISCUSSION

Mr. L. A. Shipman[1] *(presented in written form)*.—I wish to quote from the Carnegie Institute of Technology's *Contribution No. 45*, by B. Juettner: "Carbonization of coal involves the loss of volatile decomposition products such as gas, tar and water, and the formation of a condensed, non-volatile residue. The more important factors influencing the degree of condensation of the residue are: the final temperature, the rate of heating, and the nature of the coal. Another factor may be the speed at which the decomposition products are removed, which in turn depends largely on the thermal variables mentioned above, on the pressure in the retort during carbonization and on the particle size of the coal. The ash of the coal may also exert an influence due to catalytic effects."

William B. Warren at the same research laboratory has conducted numerous experiments demonstrating the effects of the rate of heating and of final maximum temperature in the carbonization of several bituminous coals. He has demonstrated that at low rates of heating at the time the coal is (in his phraseology) in the "sensitive range," some of the liquid products condense and may act as solvents on the coal becoming larger heavier molecules. Mr. Warren summarizes his results on varying the rates of heating as follows: "It seems probable that there is for each coal a maximum potential yield of distillation products which is obtainable when the rate of heating is so high that no condensation of the depolymerized molecules takes place while the temperature of the coal is within the sensitive range. Higher rates of heating would have no further effect. On the other hand, there would be some minimum yield of distillation products which is obtained where the heating rate is so low that the greatest possible amount of condensation takes place while the temperature of the coal is within the sensitive range and reducing the rate below that at which complete condensation takes place will result in no further decrease in yield of distillation products."

The point I wish to bring out is that there is no such thing as the gas- and coke-making property of a coal unless all physical and chemical conditions surrounding such tests are standardized. By varying these physical and chemical conditions of the test, the composition of the gas and tar and the quality of the coke can be changed from the same coal.

I do not wish to belittle any efforts made to perfect apparatus or test procedures. The ultimate goal of all these laboratory tests is a practical check on industrial apparatus. They have been developed so that the physical and chemical conditions of the test and apparatus would give results as nearly as possible comparable with industrial results in order to compare different coals. Much has been learned by this procedure and many new developments have resulted from it. As far as applying these procedures to tests for coal to be used on stokers where a non-coking coal can be made to coke

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or mat and a coking coal can be made to burn with a free open fuel bed, the physical and chemical conditions surrounding the combustion in a stoker fuel bed can be varied so many ways that only the extreme indications as to coking or non-coking values as set up on these tests can be of value.

MR. P. NICHOLLS.[2]—The process of caking and coking in fuel beds differs from that in coke ovens in that there is the added factor of air or products of combustion passing around the pieces of coal, and that there is burning of the volatile matter or oxidation of the surfaces. We have shown that the amount of caking and the size of the pieces of coke are dependent on the air rate. If it is high enough, caking is prevented at those interstices through which air can pass. At low air rates the fusion of the coal can close the free spaces soon enough that the coking of the mass of coal should follow the process in an oven, but if the rate is high enough the air can burn the tar and prevent closing of the openings; thus one would expect little direct correlation with the various coking tests.

Studies of fuel beds are not well suited to give numerical measures of the relative caking and coking properties of coals. Recently we used a method which, briefly, consisted in producing a standard hot bed of high-temperature coke; on this a given weight of the coal, closely sized, was fired. Only primary air at a constant rate was supplied. Four measures of the caking and coking were possible: first, a time plot of the pressure drop across the bed and its maximum value; second, the time elapsing for the appearance of flame at the sides of the furnace, and of a red spot at the center of the bed; third, the average rate of gasification for a given weight of combustible matter lost; and fourth, the fuel bed was dropped when a prefixed weight of combustible remained unburned; when cooled the coke residue was screened to determine the weights of the various sizes. Details of the method could be improved but the results were satisfactory.

In spite of there being this additional factor, characteristics of caking and coking as determined in a nonoxidizing atmosphere will be useful in studies of fuel beds. Close coordination cannot be expected because there is the additional variable of the rate of heating.

MR. J. D. DAVIS.[3]—Mr. Malleis, in his criticism of the agglutinating value test, may have had reference to some test results that we have obtained, and perhaps others, where it did not make any difference on the agglutinating value what proportion of low or high volatile coals was blended. I have about ten cases that prove that statement. Mr. Malleis is correct. You get the same results whether you have 20 per cent low volatile or 30 per cent, so it does not help us on blending. However, it does mean something. For one thing, in our study of the characteristics of coal constituents of the same bed, we can distinguish pretty sharply by the agglutinating value the amount of splint as distinguished from bright coal in the same bed. The splint usually, though not always, has a much lower agglutinating value than bright coal. We have one case on record where a splint layer actually had a higher agglutinating value than bright coal, so it does not hold universally even in that respect.

MR. R. A. SHERMAN.[4]—On the third page of his paper, Mr. Malleis makes the

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[3] Chemist, U. S. Bureau of Mines, Pittsburgh, Pa.

[4] Fuel Engineer, Battelle Memorial Institute, Columbus, Ohio.

statement: "Low-volatile coals when heated in the standard volatile-matter test, usually expand to many times the volume of the original coal used for the test, and often the resulting coke button will completely fill the test crucible." This observation agrees with the general conception of the coke button obtained from low-volatile coals. I should like to offer some data which present a different picture.

In writing a report of some recent tests on the burning of coal on small stokers, we wished to have some simple indication that would give the laymen a conception of the difference in the coking properties of the four coals. The coals, which were Millers Creek, Illinois, Pittsburgh, and Pocahontas, were so widely different that we felt the volatile-matter coke button would show what we wanted. When we made the buttons in the standard electric furnace, we found that those from Pocahontas coal were small dense buttons instead of the large button that is normally expected. We made a number of buttons from various samples of the same lot of coal and found that they were all alike.

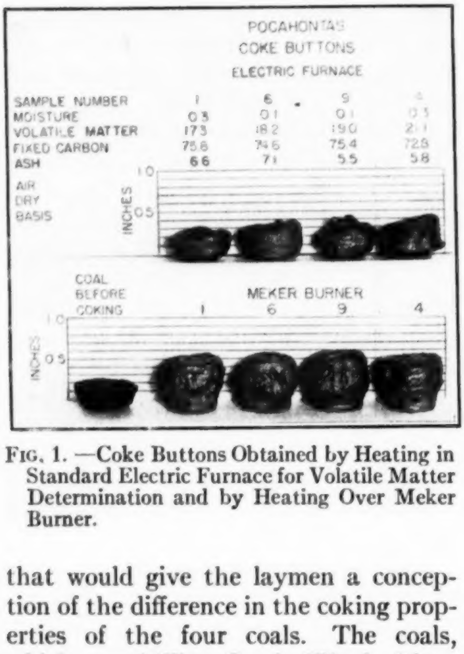

FIG. 1.—Coke Buttons Obtained by Heating in Standard Electric Furnace for Volatile Matter Determination and by Heating Over Meker Burner.

A preliminary draft of the report was sent to a number of men for review, and several questioned the results that we had obtained on the Pocahontas coke button. They thought that the Pocahontas may have oxidized, as it had been in storage in the laboratory for some months. To clear up this possibility, we obtained, through the courtesy of W. E. E. Koepler, Secretary, Pocahontas Operators' Assn., samples from nine mines in the Pocahontas No. 3 field, including the same mine from which the original sample had come, samples from neighboring mines, and others at some distance away.

We ran coke buttons on these samples by the same method used before, that is, by plunging the crucible into the electric furnace at 950 C. The coke buttons were all of the same type as obtained with the original coal, unswollen buttons of hard coke.

The only reference in the literature that includes photographs of coke buttons from the standard volatile-matter test is the paper by Rose and Sebastian presented before the American Institute of Mining and Metallurgical Engineers.[5] They stated that the standard volatile-matter test showed swollen buttons. On checking with Mr. Rose, I found that the Koppers Laboratory had used at that time the Meker burner for heating the crucibles. We had suspected that, for in the meantime we had experimented by preparing some buttons in the flame of the Meker burner and also by the method of preheating the coal in the top of the electric furnace.

The accompanying Fig. 1 shows the difference in the type of buttons obtained when the coal is heated by the two methods; the results on four of the nine Pocahontas No. 3 coals are shown. There is a vast difference in the type of

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[5] H. J. Rose and J. S. Sebastian, "Changes in Properties of Coking Coals Due to Moderate Oxidation During Storage," *Transactions*, Coal Division, Am. Inst. Mining and Metallurgical Engrs., Vol. 88, p. 556 (1930).

button obtained. It would appear that the electric-furnace method is somewhat better for preparation of the buttons, as with the gas burner all of the buttons swelled to the top of the crucible and little distinction is seen among the buttons.

I have recently learned from Mr. Fieldner that they had had similar experiences in the difference in the type of button obtained. If we had consulted him, we would have avoided the necessity of finding out for ourselves. Inasmuch as the effect of the difference in the type of heating was not clear to several with whom we discussed the problem, it seemed worthy of presentation so that others would be spared this difficulty.

MR. B. P. MULCAHY.[6]—I think the point which Mr. Sherman brought out about the effect of temperature, particularly in the case of this volatile determination, is quite important. You can take like samples of the same coal, subject these samples to the volatile-matter test but alter the temperature of testing by increments of 100 deg. Cent. from 500 C. to 900 C. and obtain different sized buttons for each temperature.

There are one or two other points which should be emphasized, I think: The first is the evaluation of the plastometer test. I do not know how much importance is generally attached to this test, but our concept of the coking process takes cognizance of this plastic range of coal. As we visualize this picture of coking we feel that the rate of gaseous release from the coal at that particular stage has a great deal to do with the ultimate structure of the coke. We, as coke manufacturers, pin a lot of hope to some of these plastic range determinations, for we feel that it will ultimately relieve us of the very arduous job of evaluating a coal by subjecting it to full-scale oven tests.

One point Mr. Malleis brought out was his regret that these laboratory appraisal tests of coal actually show nothing at all about the value of the coke. The seriousness of this contention is realized when you consider that anywhere from 70 to 80 per cent of the coal as charged comes out as coke and that these laboratory tests really evaluate the by-product yield and nothing else.

There are, however, certain tests which look as if they might point the way out of this haze. One is the expansion tester recently developed which we personally feel is going to show us a good deal and I know, too, that as producers we certainly owe a debt to the U. S. Bureau of Mines and Mr. Davis particularly for the work they have been doing on coal at Pittsburgh and have published in *Monograph No. 5.*

We have always felt that, unfortunately, from a coke manufacturer's standpoint the attack on some of these problems was almost too fundamental to be of definite value to us. Work of a fundamental nature should be very definitely encouraged, but we have to remember, too, that we must keep our feet on the ground when we consider this fundamental work, and at the same time broaden our concepts by discovering the relationships with what we encounter in actual practice. If this will be our attitude I believe that we can make greater progress in coal carbonization.

MR. C. C. RUSSELL.[7]—I have been particularly interested in listening to all this discussion in view of the work of the new subcommittee, of Committee D-5, on plasticity and swelling of coal. As you may know, this subcommittee was formed a year ago and we have had two

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[7] Engineering and Construction Division, Koppers Co., Kearney, N. J.

meetings in which the work has been started. The purpose of the subcommittee is to study the methods which have been proposed for the determination of both plasticity and expansion of coal. For some time at least there is no thought of trying to standardize any one method. We have included the study of methods for the determination of the expansion of coal because of the large amount of interest in the subject and because it is so closely related to the plastic state of coal.

There still appears to be considerable divergence of opinion concerning the measurement of plasticity of relatively homogeneous materials so the measurement of the plasticity of coal—a most heterogeneous material—is a very difficult one.

There is one thing about this matter of coal expansion that might be well to mention. Certain coals expand and are dangerous to charge into coke ovens alone. However, when these coals are mixed with contracting coals there are certain mixtures that contract and are consequently perfectly safe in the ovens. Coke oven operators usually know these badly expanding coals and would never charge them alone into their ovens. These coals may, however, be very dangerous even in mixtures, for in large-scale operation the mixtures are not always homogeneous, or there may be segregation after mixing.

I wish to thank Mr. Malleis for his excellent summary. It will be a great help in our committee work.

MR. CARL D. ULMER.[8]—While Mr. Malleis has given much careful thought to his subject and has written an excellent summary of existing procedures, his review surveys only the American literature, and the methods he describes are those used generally only in this country. It would be not only interesting but also instructive had the paper included brief mention of and references to laboratory tests recently developed abroad. One recent survey of tests now employed in Germany for determining the products obtainable from coal by carbonization, known to few American fuel technologists, is the doctoral dissertation by Gerhard Lorenzen entitled "Beiträge zur Untersuchung von Kohlen auf ihre Verkokbarkeit und zur Herstellung von Mitteltemperaturkoks" presented to the Technischen Hochschule, Berlin, in 1935.

MR. O. O. MALLEIS[9] (*author's closure, by letter*).—The discussion further confirms the complexity of the problems of developing and standardizing methods of test for determining the relative caking, plastic, gas- and coke-making properties of bituminous coals.

Undoubtedly most of the interest in these methods of test will continue to be with regard to their applications in the evaluation of coal for the manufacture of coke. It is hoped, however, that more interest may be stimulated in the future with regard to the possible application of some of these methods of test with regard to caking and coking characteristics of bituminous coal in the fuel beds.

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[9] Manager, Inspection Division, Appalachian Coals, Inc., Cincinnati, Ohio.

SIGNIFICANCE OF FRIABILITY AND SIZE STABILITY TESTS ON COAL[1]

By R. E. Gilmore[2] and J. H. H. Nicolls[2]

This paper deals primarily with the significance of the A.S.T.M. "Proposed Method of Tumbler Test for Coal" and "Proposed Method of Drop Shatter Test for Coal."[3] The standardization of these test methods was undertaken by Subcommittee XI on Coal Friability, of the Society's Committee D-5 on Coal and Coke. The work of this subcommittee has been in progress since 1932. The comparative friability tests leading up to their adoption have been fully reviewed in a recent publication[4] and need only to be outlined and briefly discussed here.

By friability, as applied to coal, is meant its liability to break or crumble into smaller pieces when subjected to handling; hence a friability test serves to determine relative handling properties. Size stability is just the opposite of friability and implies resistance to breakage. When examined by a laboratory test method the friability of a coal may be expressed as "friability, per cent," which is the average size of the coal resulting from breakage during the test, as a percentage of the average size of the lumps taken as sample. The difference between one hundred and the friability per cent is the "size stability, per cent": that is, these values are complementary to each other.

The most comprehensive method of ascertaining the handling properties of a coal is to examine shipments by screening at different stages during its mining, preparation, and transportation from the mine to its ultimate consumer. Testing and comparing coals by this method is too costly and, therefore, impractical, and what has been required is a laboratory method, or methods, to serve as a definite measure of the friability of coals. In the development of laboratory friability methods, the principle generally followed has been that of performing a uniform amount of work on the sample, and then measuring the reduction in average size of the lumps.

Seven different methods, five tumbler and two shatter, were tried out in the experimental work of the friability subcommittee, and an endeavor was made to ascertain to what extent the apparatus and methods previously adopted as A.S.T.M. standards for coke would be suitable for coal. Results obtained with seven different standard coals varying from hard non-friable anthracite to soft, quite friable, bituminous coals, showed that certain variations of all seven methods could be considered satisfactory in that they placed the seven coals in approximately the same order as to friability. On the assumption that there was a need for two methods, one

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[1] Published by permission of Director of Mines and Geology Branch, Department of Mines and Resources, Canada.

[2] Senior Engineer, and Chemist, respectively, Fuel Research Laboratories, Division of Fuels, Canadian Bureau of Mines, Ottawa, Ont., Canada.

[3] A. S. T. M. Standards on Coal and Coke—Prepared by Committee D-5, September, 1936, pp. 70 and 63, respectively. In this publication the two methods of test are given as Proposed Drafts. These two proposed methods were approved this year for publication as tentative standards, see *Proceedings*, Am. Soc. Testing Mats., Vol. 37, Part I, pp. 820 and 827 (1937).

[4] R. E. Gilmore, J. H. H. Nicolls and G. P. Connell, "Coal Friability Tests," Mines Branch *No. 762*, Department of Mines, Canada.

tumbler and one shatter as with coke, these were selected from the seven.

The purpose and scope of the two methods selected[3] may be abstracted as follows:

The tumbler test for coal is intended for determining the relative friability of a particular size of lump coal. It affords a means of measuring the liability of coal to break into smaller pieces when subjected to repeated handling at the mine, or subsequently by the distributor or by the consumer. It also may serve to indicate the relative extent to which lump coals will suffer size degradation in certain mechanical feed devices. Furthermore, the test may be employed for differentiating between certain ranks and grades of coal, and hence be of service for coal classification purposes.

TABLE I.—COMPARISON OF "TUMBLER" AND "DROP SHATTER" TESTS FOR COAL ACCORDING TO A.S.T.M. METHODS.

	Tumbler Test[3] (on 1.05 to 1.5 in. square hole size lumps)	Drop Shatter Test[3] (for 2 to 3 in. round hole size lumps)
Apparatus	Porcelain jar tumbler such as employed for pulverizing coal samples—or optional iron jar tumbler—fitted with iron frame and lifting shelves as illustrated in Fig. 1.	Shatter test machine as illustrated in Fig. 1, and in the Standard Methods of Shatter Test for Coke (D 141-23).[5]
Screens	Square mesh screens, having 1.5, 1.05, 0.742, 0.525, 0.371, 0.0469 and 0.0117-in. openings.	Round-hole screens, having 3, 2, 1½, 1, ¾, and ½-in. openings.
Collection of gross sample	In accordance with Sections 1 to 4 of A.S.T.M. D 21-16.	In accordance with Sections 4 and 5 of A.S.T.M. D 410 - 35 T.
Preparation of sample for test	Following preliminary screening of representative portion of gross sample 25 lb. of 1.05 to 1.5-in. lumps are prepared.	Following preliminary screening of representative portion of gross sample 125 lb. of 2 to 3-in. lumps are prepared.
Procedure	1000-g. of rescreened 1.05 to 1.5-in. lumps tumbled in jar-mill tumbler test machine at 40 r.p.m. for 1 hr.—screen analysis then made on tumbled coal.	50 lb. of rescreened 2 to 3-in. lumps dropped twice in Shatter test machine—screen analysis then made on dropped coal.
Recording of results	Results tabulated and calculated as per numerical example.	Results tabulated and calculated as per numerical example in Table I.
Results to be reported	Results reported as "Friability per cent"; $\frac{100\,(S - s)}{S}$ where S is the average size of the lumps taken as sample, and s is the average size of the tumbled coal.	Results reported as size stability per cent; $\frac{(s) \times 100}{(S)}$ where S is the average size of the lumps taken as sample and s is the average size of the dropped coal.
Supplementary reportable data	"Dust Index" representing fines and dust produced in the test, and indicative of proportion of breakage caused by abrasion.	"Slack Index"—the per cent of the dropped coal passing the ¾-in. screen—indicative of the comparative slack-producing characteristics of the coal tested.

The drop shatter test for coal is intended for measuring the relative size stability, and its complement the friability, of lump coal. It affords a means of indicating the ability of coal to withstand breakage when subjected to handling at the mine and during its transit to the consumer. The method of test is considered applicable for testing both a standard size of different coals and for testing different sizes of the same coal, 2 to 3 in. being recommended as the standard size for testing different coals.

The drop shatter test method includes separate procedures for testing the 2 to 3-in. standard size of lumps, and for testing other sizes (both single and mixed sizes). Quoting further on the scope for this test:

The method appears best suited for measuring the relative resistance to breakage of the larger sizes of lump coal when handled in thin layers such as from loader to mine car, from loading boom to railroad car, from shovel to chute, etc. While it may not be so well adapted for measuring the liability to breakage of coal when handled in mass, as in unloading open bottom cars, emptying bins, etc., it is believed that the method of test and particularly the supplementary method will serve to indicate the relative size stability of composite sizes of coal where, in commercial handling, the smaller sized particles and lumps have a cushioning effect which tends to lessen the breakage of the larger lumps of coal.

A comparison of these two test methods is given in Table I.

It is to be noted that the tumbler test, in which 1000 g. (2.2 lb.) of lumps of coal are used as individual samples, is a small-scale laboratory test, and that the drop shatter test, requiring 50-lb. samples of different sizes of coal, may be described as a large-scale laboratory test. A second noteworthy difference is in respect to size of lumps used as sample and the kind of screens used in their preparation. In the tumbler test 1 to 1½-in. lumps prepared on square-hole screens are specified, whereas the drop shatter test calls for 2 to 3 in. and other size lumps prepared on round-hole screens. Furthermore, it is to be observed that, while the proposed drop shatter test for coal is similar in many respects to the shatter test for coke, Standard Methods of Shatter Test for Coke (D 141 – 23),[5] the tumbler test for coal is quite different from the Standard Method of Tumbler Test for Coke (D 294 – 29).[6]

Shattering versus Abrasion:

The breakage taking place in the laboratory test is caused both by the shattering effect of the lumps falling either on a hard surface or on one another, and by abrasion due to the lumps rubbing against one another. In the tumbler test where the lumps fall only short distances in the small porcelain (or iron) jar used, and where they continually rub against one another, the breakage caused by abrasion is much more emphasized than in the drop shatter test where the lumps drop a distance of 6 ft. and rub against one another very little. On a given coal the total breakage taking place in the tumbler test, as indicated by the calculated friability per cent, is appreciably more than in the drop shatter test. The procedure adopted in the latter test, of dropping a 50-lb. sample twice in succession, was considered to produce a breakage effect representing that taking place in the ordinary commercial handling of coal, whereas the tumbler test serves to determine the inherent weakness of lumps of coal when subjected to repeated handling.

DUPLICABILITY OF TESTS

The duplicability of tests is presented under three headings: tumbler test for coal, drop shatter test for coal—standard 2 to 3-in. lumps, and supplementary drop shatter test method for other sizes. Data involved are abstracted from those already published[5] covering comparative friability tests conducted up to the end of 1934, and from experimental results obtained and on record since then at the Fuel Research Laboratories, Ottawa.

Tumbler Test for Coal:

The procedure for this test stipulates that "at least four single-jar tests shall be made and, provided sufficient sample is available, it is recommended that two or more four-jar tests be made" and that "when two or more four-jar tests are made the contents of the four jars from each set may be mixed and screened together." It is, however, allowed that "when only four single-jar tests are made, the contents of each jar shall be screened separately, in order to be sure that there is satisfactory agreement between the results obtained." A numerical example, serviceable for either a single-jar or a four-jar test, complete

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[5] 1936 Book of A.S.T.M. Standards, Part II, p. 449.

[6] *Ibid.*, p. 453.

with the method employed for tabulating the screen analysis of the tumbled coal and for calculating and recording the friability per cent value is given at the end of the proposed tumbler test.[3]

Using some results recently obtained at the Fuel Research Laboratories on the 1 to 1½-in. square mesh lumps of two lots of coal, A—Welsh anthracite, B—Westphalian anthracite, comparison of the duplicability of single-jar tests with that of four-jar tests may be made here:

FRIABILITY, PER CENT

SINGLE-JAR TESTS

1-hr. tumbling:						
Coal A..	25.4	26.1	22.0	....	(4.1 Range)	(24.5 Avg.)
Coal B..	30.4	35.1	29.9	27.8	(7.3 Range)	(30.8 Avg.)
3-hr. tumbling:						
Coal A..	40.4	43.2	38.7	34.1	(9.1 Range)	(39.1 Avg.)
Coal A..	44.2	44.1	39.7	....	(4.5 Range)	(42.0 Avg.)
Coal B..	43.9	42.5	41.8	....	(2.1 Range)	(42.5 Avg.)
Coal B..	43.3	44.6	43.2	43.1	(1.5 Range)	(43.5 Avg.)

FOUR-JAR TESTS

3-hr. tumbling:						
Coal A..	39.1	40.5	41.6	....	(2.5 Range)	(40.4 Avg.)
Coal B..	44.7	43.5	41.7	....	(3.0 Range)	(43.3 Avg.)

These results demonstrate that closer checks are obtained in two or more four-jar tests than in a set of four single-jar tests. This is to be expected since not only is the experimental error lessened when screening the contents of four jars at one time, but the amount of the sample used is two and three times larger when making duplicate and triplicate four-jar tests than in making a set of four single-jar tests.

Comparison of 1 and 3 hr. as Time of Tumbling.—The tumbler test as tentatively adopted specifies 1 hr. as the duration of the actual tumbling test in the jar or jars, rotated at 40 r.p.m. A 3-hr. test procedure was previously used in making tests at the Fuel Research Laboratories, at the Seattle Experiment Station of the U. S. Bureau of Mines and at West Virginia State University. Hence a comparison of the duplicability of the results obtainable by the different times of tumbling may well be further reviewed here. For this purpose the results given in the paper on Coal Friability Tests[4] and those shown above for Welsh and Westphalian anthracites may be used as below:

	Friability Values, per cent		
	1-hr. Tumbling	3-hr. Tumbling	Ratio, 3-hr. to 1-hr. Results
No 1—Pennsylvania Anthracite........	19	27	1.4
No 2—Welsh Anthracite..	27	42	1.6
No. A—Welsh Anthracite..	26	40	1.5
No. B—Westphalian Anthracite........	31	44	1.4
No. 3—Pennsylvania Bituminous........	25	39	1.6
No. 4—N.S. Bituminous...	28	40	1.4
No. 6—B.C. Bituminous..	59	70	1.2
No. 7—B.C. Bituminous..	42	55	1.3

The average ratio to the second decimal for the above eight coals was 1.42, and the average ratio for thirty other coals ranging in rank from lignite to anthracite was found to be 1.41. Hence 1.4 may be kept in mind as the average ratio of the friability per cent values for the 3-hr. tumbling tests to those for the 1-hr. tests. Eliminating the 1.2 and the 1.6 ratios in the above list, and two other low and two high values in the thirty coals does not alter the 1.4 figure. This average, however, is general only, since with the low and high values eliminated the ratio varied roughly from 1.31 to 1.49.

The differences between the individual maximum and minimum friability values for the thirty coals just mentioned ranged from 0 to 4.2 per cent for the 1-hr. tests and from 0.1 to 3.5 per cent for the 3-hr. tests. After eliminating four differences greater than 3.5 per cent in the 1-hr. results, the average difference between the maximum and minimum was the same for the 1-hr tests as for the 3-hr. tests, that is, 1.5. The results indicate that as good checks were obtained with the very friable as with the medium and non-friable coals. This confirms the assumption that for practical purposes the 1-hr., as specified in the tentative tumbler test method, is as good in respect to duplicability of tests as the 3-hr. tumbling which has been used extensively up to the present.

In accordance with the above, the authors now wish to recommend that omission of directions as to closeness of two or more results in the tumbler test[3] be corrected, and that to the end of "Procedure" (Section 8) be added the following:

> At least four single-jar tests shall be made. When there is insufficient coal for eight individual jars the contents of each jar shall be screened separately. Any calculated friability per cent value which is not within 5 per cent of some other value shall be rejected and the remaining values averaged. When there is sufficient coal for eight or more individual jars the tests shall be considered as four-jar tests and the contents of a group of four jars screened together. Tests on a given sample shall be repeated until at least two (four-jar) friability results shall be obtained which agree within 2 per cent, and when three or more such four-jar tests are made any value which is within 3 per cent of another value shall be included in the average results to be reported.

Effect of Using 1.25-in. Screen.—On the assumption that the variation in friability per cent values obtained in successive tests on a given coal was probably due to non-uniformity of the size of lumps in the sample, a series of tests was made on 1.05 to 1.5-in. lumps in which the proportion larger than 1.25 in. varied by increments of roughly 20 per cent from 90 to 10 per cent. In this series a 1.25-in. screen was used in preparing the coal for tumbling and in the screen analyses after tumbling. A medium friable coal was used and the following average results were obtained. The first of each pair of friability per cent values was calculated by the usual procedure, in which any values obtained with the 1.25-in. screen are disregarded on the assumption that the coal as tested had an average diameter of 1.25 in. and that all the coal remaining on the 1.05-in. screen after tumbling had a corresponding average diameter. The second of each pair of values was calculated by employing the values obtained with the 1.25-in. screen in addition to those with other screens both before and after tumbling.

AMOUNT OF COAL ON 1.25-IN. SCREEN	FRIABILITY OBTAINED WITHOUT AND WITH USE OF 1.25-IN. SCREEN VALUES IN THE CALCULATIONS, PER CENT	
	WITHOUT	WITH
1-hr. tests		
70 per cent....	33.1	34.4
50 per cent....	39.0	39.3
3-hr. tests		
90 per cent....	49.9	54.9
70 per cent....	48.1	50.5
50 per cent....	48.3	49.4
29 per cent....	52.8	51.7
11 per cent....	52.7	51.3

The 1-hr. single-jar tests made in pairs showed respective friability averages of 33.1 and 39.0 per cent by the ordinary method of calculation for samples in which the proportions of the 1.25-in. lumps were 70 and 50 per cent, respectively. These values were increased, it will be noticed, to only 34.4 and 39.3 per cent using the 1.25-in. screen. The 3-hr. single-jar tests, made usually in fours and employing coal averaging in size from 90 to 10 per cent on the 1.25-in. screen, gave friability values ranging from 48.1 to 52.8 by the ordinary method of calculation, the largest of which were obtained with the smaller size coal. These values were changed by amounts varying from 1 to 5 per cent by using the 1.25-in. screen. The greatest change was obtained with the large coal, and this so altered the friability per cent values as to transfer the highest value from the small to the large coal.

Since the values obtained with the 1.25-in. screen must be more reliable than those obtained by the usual method of calculation, it follows that there will probably be less liability to error if the smaller sizes within a size range of coal predominate than if the large sizes do so. The intermediate sizes of coal, ranging from 70 to 30 per cent on 1.25-in. screen, gave friability values from 48.1 to 52.8 or a range of 4.7 by the usual method of calculation, and values from 49.4 to 51.7 a range of 2.3 only by the method employing the 1.25-in. screen. However, in series of duplicate, triplicate and quadruplicate tests, the individual friability per cent values do not check any more closely when the 1.25-in. screen is used than when it is not employed in the calculations.

The foregoing data point to the advisability of using coal prepared in such a way that it will uniformly include sizes covering the whole range between 1.05 and 1.5 in., as provided in Section 6(*c*) of the A.S.T.M. proposed tumbler test method for coal.[3]

Dust Index.—As given in the footnote of the numerical example shown in Section 9 of the proposed tumbler test, the "dust index" is the percentage of the tumbled coal passing 0.0117 in., equivalent to Tyler 48 mesh. This index represents the proportion of the breakage due to abrasion, or attrition, rather than to shattering. The proportion of breakage due to abrasion ranges from approximately one-quarter to one-half of the total breakage taking place in the 1-hr. tumbling test, as indicated by the friability per cent, and fairly close check results are obtainable in duplicate tests on individual coals. The percentage of the tumbled coal in the standard 1-hr. tests through the 48-mesh screen varies from about 8 for non-friable anthracite to as high as 35 for very friable bituminous coals. This value was found to vary from 13 to 30 for fifteen medium friable coals and was not uniformly proportional to their friability per cent values. The percentage through the 0.0029-in. (200-mesh) screen was taken as an index of the dust-forming capacity of the coals tested in the friability test as originally developed,[7] which 200-mesh index has not been found to be of any more value than the 48-mesh index.

The development of a method to test the dust-forming propensities of coal as undertaken by Subcommittee XIV on Dustiness of Coal and Coke of the Society's Committee D-5 on Coal and Coke may be referred to here. This method is a direct determination of the dust formed when a given quantity of lump coal is dropped in a closed chamber and therefore may be more practical for the determination of dustiness than the tumbler test.

If such proves to be the case, the term "dust index" in the tumbler test may well be changed to "abrasion index" since the percentage through 0.0117-in. (48-mesh) screen serves for differentiating between coals that produce a large amount of fines and dust by abrasion and those which, although they show the same friability per cent value, produce a much smaller amount of fines due to their inherent toughness. Examples of this are Pocahontas low-volatile, coking bituminous coal and Alberta high-volatile, non-coking bituminous coal showing approximately the same friability per cent but with percentages of tumbled coal through the 48-mesh screen of 28 and 15, respectively.

Non-Friable, Medium Friable, and Very Friable Groups.—At this point an attempt will be made to designate what is meant by the terms non-friable, medium friable and very friable coals. From the results on record at the Fuel Research Laboratories the following are

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[7] By J. H. H. Nicolls, "Friability Tests on Various Fuels Sold in Canada," Mines Branch *No. 644*, Department of Mines, Canada (1926).

suggested limits for grouping coals in respect to friability as tested by the 1-hr. tumbler test:

FRIABILITY, PER CENT	
Below 20....	*Non-friable coals*—including mainly the hard non-friable varieties of anthracites.
20 to 30.....	*Medium friable A coals*—including the softer varieties of anthracites, the harder varieties of bituminous, and the tough varieties of sub-bituminous coals.
30 to 40.....	*Medium friable B coals*—including the softer varieties of anthracites, and the less hard varieties of medium and high volatile bituminous coals.
40 to 50.....	*Friable coals*—including mainly the softer varieties of low, medium, and high volatile bituminous coals.
Above 50....	*Very friable coals.*

This grouping, which is presented tentatively and subject to revision, the authors trust will serve as a basis for constructive criticism by fuel technologists generally, and particularly those interested in coal friability.

Drop Shatter Test for Coal—Standard 2- to 3-in. Lumps:

Results of a series of tests made on the 2 to 3-in. lumps of coal 4A—a medium friable bituminous coal from Nova Scotia, reported in the paper on Coal Friability Tests[4] may be summarized as follows:

	TEST No. 1	TEST No. 2	TEST No. 3	TEST No. 4
Number of 50-lb. samples tested..	one	two	two	two
Percentage of sample on 2½-in. screen..........	...	....	28.8	37.0
Amount of dropped coal screened, lb.	50	100	100	100
Dust through 48-mesh screen and loss by difference, per cent........	0.4	0.3	0.3	0.3
Size stability, per cent...........	81.4	81.5	81.8	83.1

Further comparative results on the 2 to 3-in. standard and on smaller sizes of three coals varying widely in friability, as reported for coals 1A—Pennsylvania anthracite and 7A a "friable" high-volatile bituminous coal, in addition to coal 4A mentioned above, are given below:

Coal and Size Designation	Size Stability Values, per cent		
	One Lot of 50 lb. Dropped and Screened	Two 50-lb. Samples Dropped with Screening Test on Combined 100 lb. of Dropped Coal	Difference
1A — Pennsylvania anthracite			
Standard 2 to 3-in. lumps.....	93.9	92.8	1.1
1½ to 2-in. size..............	93.5	93.2	0.3
1 to 1½-in. size..............	95.0	95.1	0.1
4A — Medium friable (Canadian) bituminous coal			
Standard 2 to 3-in. lumps.....	81.4	81.5	0.1
1½ to 2-in. size..............	85.7	85.1	0.6
1 to 1½-in size..............	90.2	90.0	0.2
7A — Friable (Canadian) bituminous coal			
Standard 2 to 3-in. lumps.....	64.4	67.9	3.5
1½ to 2-in. size..............	70.4	73.4	2.0
1 to 1½-in. size..............	79.7	81.1	1.4

These results show that, while check results within 1.5 size stability per cent values were readily obtained on non-friable and medium friable coals, the variance in the corresponding results on the very friable coal was 3.5. In the experimental work affording these results it was noted that, whereas generally speaking closer check results were obtainable with the sizes smaller than the standard 2 to 3-in. lumps, the duplicability of tests on this standard size was appreciably better than on the larger 3 to 4-in. lumps also tested.

The ratio of round hole to square hole for coal was found to average 1.14 and the ratio of square hole to round hole its reciprocal, 0.88. Using this ratio, the 2-in. square mesh opening adopted as the limiting size (that is the lower limit) for preparing the sample for the shatter test for coke (D 141)[5] corresponds roughly to 2¼-in. round-hole screen opening, for lump coal; and the 3-in. square-mesh opening of the upper limiting screen size adopted in the tumbler test for coke (D 294)[6] corresponds to slightly less than 3½-in. round-hole opening. Hence the 2 to 3-in. round hole size specified for lump coal in the drop shatter test is smaller than the size of the lumps used in either the tumbler or shatter test for coke, but is appreciably larger than the 1 to 1½-in. square mesh size specified in the tumbler test for coal.

Effect of Varying Preparation of Sample.—The results of two series of tests made to ascertain the effect of variation in preparing the 2 to 3-in. standard size of lump coal are of interest at this point. These tests, which were made on two medium friable high volatile bituminous coals, on coal 4B from the Sydney area, Nova Scotia, and on coal C from the Pittsburgh seam, Westmoreland County, Pennsylvania, comprised drop shatter tests on the 2 to 3-in., lumps obtained by screening out this size (1) from a supply of screened run-of-mine coal in the storage bin, (2) from larger lumps, that is, larger than 3 in. after dropping them in the shatter test machine, and (3) from larger lumps broken down with a hammer. The size stability results to the nearest 0.5 obtained in successive tests where the dropped coal from two 50-lb. samples was screened as one lot are hereunder summarized:

Coal 4B—Nova Scotia High Volatile Bituminous.

		AVERAGE
First	Four tests on lumps obtained by screening supply from storage bin showed size stability per cent values of 79.0, 80.0, 78.0, 79.0	79.0
Second	Two tests on lumps obtained from larger lumps after dropping in shatter test machine—77.5 and 80.0	79.0
Third	Three tests on lumps obtained from larger lumps by hammer 78.5, 80.0 and 77.0	78.5

Coal C—Pennsylvania High Volatile Bituminous.

		AVERAGE
First	Seven tests on lumps obtained by screening supply from storage bin showed size stability per cent values of 89.0, 88.5, 88.0, 88.0, 86.0, 88.0, 87.5	88.0
Second	Three tests on lumps obtained from larger lumps after dropping in shatter test machine—87.0, 88.5 and 90.5	88.5
Third	Three tests on lumps obtained from larger lumps by hammer, 87.5, 88.5, 87.5	88.0

This shows that size stability results checking within 2 per cent are readily obtainable on the 2 to 3-in. standard size and that freshly broken lumps obtained from larger lumps, either by dropping them in the shatter test machine or by reducing them with the hammer, are satisfactory for augmenting the sample from a given lot of coal. The handling during the preparation of the gross sample and augmenting the sample for test as just described is beneficial for discarding lumps in which fracture lines make them abnormally weak. In this connection care should be taken to follow closely the directions specified in Section 6 of the proposed method in respect to "rescreening of sample for test," and especially to the directions reading "individual pieces of coal not passing readily through either of the screens shall be tried by hand to see if they will pass through the openings in any position without forcing."

This point is also important for screening the dropped coal on the larger 2-, 1½ and 1-in. screens. Furthermore the directions concerning the weighing of the different screen sizes of the dropped coal are to be stressed. As described in Section 7(*b*) of the published method, this is best carried out by the "cumulative operation method" in which the 2 to 3-in. lumps are weighed into a tared container, which is then reweighed after addition of the successive individual smaller screened sizes of dropped coal.

Variation of Procedure for Screening Dropped Coal.—Most of the routine tests conducted at the Fuel Research Laboratories are made using a variation of the published procedure for screening the dropped coal. This is done by the cumulative weighing method, but on 100 lb. resulting from dropping two successive 50-lb. lots of the coal being tested, instead of on each 50 lb. of dropped coal. This variation, which is in accordance with the alternative directions for "larger size lumps" given in Section 14 of the drop shatter test, under the supplementary method procedure for testing other "single" sizes, is to be recommended for use when testing the standard 2 to 3-in. lumps.

A numerical example for recording and calculating the results of the drop shatter test on a single 50-lb. sample is given in Section 7 under "Procedure" of the drop shatter test. This numerical example is also applicable for screening 100 lb. of dropped coal, when the "weight per cent" column also serves as the "weight recorded" column.

High, Medium, and Low Size Stability Groups.—Using the results already published[4] and those on record, the following are suggested as limits for grouping coals in respect to size stability as determined by the drop shatter test on the 2 to 3-in. standard size:

Size Stability, per cent	
Above 90	*High size stability coals* including mainly the hard non-friable varieties of anthracites.
80 to 90	*Medium size stability coals, subgroup A* including the medium friable anthracites, the more stable low, medium, and high volatile bituminous coals, and the tough varieties of (non-weathered) lower rank subbituminous and lignite coals.
70 to 80	*Medium size stability coals, subgroup B* including mainly the less stable low, medium and high volatile bituminous coals, and the less stable lower rank coals.
Below 70	*Low size stability coals* including the friable and very friable bituminous and lower rank coals.

This grouping is tentative only, and should be studied and constructively criticized in relation to the friability per cent grouping suggested earlier in this paper for interpreting the results of the tumbler test for coal, and in relation to the significance of the supplementary method of test for sizes other than the standard 2 to 3-in. lumps given below.

Supplementary Drop Shatter Test Method for Sizes Other than the Standard 2- to 3-in. Lumps:

Sections 9 to 15 inclusive of the "Proposed Method of Drop Shatter Test for Coal," pertain to a supplementary method of test for other "single" sizes and "mixed" sizes—the word "other" implying sizes other than the standard 2 to 3-in. lumps. By single sizes are meant 3 to 4 in., 4 to 6 in., 6 to 8 in., etc., and 1½ to 2 in., 1 to 1½ in., ¾ to 1 in., etc.—these two groups representing larger and smaller sizes of coal respectively than the 2 to 3-in. lumps. The mixed sizes comprise composites or admixtures of two or more of these single sizes.

Averages of duplicate determinations on different single and mixed sizes of coal 4A, a medium friable Canadian bituminous coal used as standard, are given in the paper on Coal Friability Tests.[4] These may be summarized as shown in Table II.

The results of multiple tests on different sizes of coal C, the Pittsburgh seam high-volatile bituminous coal, including the tests on the standard 2 to 3-in. lumps, are also of interest here, all the results given being on 50-lb. samples dropped twice with screen analyses on either single (50-lb.) or double (100-lb.) lots of dropped coal:

Lumps, in.		Size Stability, per cent	
4 to 6	Six tests	81.3, 80.3, 78.9, 78.7 76.3, 78.4	(79.0 Avg.)
3 to 4	Ten tests	84.3, 84.7, 85.8, 83.9 84.1, 85.6, 85.8, 84.1 83.0, 84.7	(84.5 Avg.)
2 to 3	Seven tests	88.0, 89.0, 88.5, 88.0 86.0, 88.0, 84.5	(87.5 Avg.)
1½ to 2	Three tests	90.2, 90.0, 92.6	(91.0 Avg.)
1 to 1½	Three tests	92.0, 92.8, 92.9	(92.5 Avg.)
¾ to 1	Two tests	94.8, 94.1	(94.5 Avg.)
½ to ¾	Two tests	96.0, 95.6	(96.0 Avg.)

Coals 4B and C affording the results just summarized, it may be repeated, were specially screened run-of-mine coal from a large supply of each coal from storage bins. These coals had received an appreciable amount of handling during their preparation, in which the 1½-in. slack had been removed, and during the special screening during the preparation of the different sizes for comparative experimental tests. This previous handling accounts to some extent for the close checking in the repeated tests on the standard 2 to 3-in. lumps and on certain of the other sizes.

TABLE II.—DATA SUMMARIZED FROM PAPER ON COAL FRIABILITY TESTS.

SINGLE SIZES, 50-LB. SAMPLES DROPPED TWICE	SIZE STABILITY, PER CENT	MIXED SIZES 50-LB. SAMPLES DROPPED TWICE	SIZE STABILITY, PER CENT
4 to 6-in. lumps	70.0	1½ to 4-in. lumps	81.8, 85.0 (83.5 Avg.)
3 to 4-in. lumps	77.0	¾ to 1½-in. lumps	91.3, 92.4 (92.0 Avg.)
2 to 3-in. lumps	83.5	Minus 4 in. coal	92.8, 94.3 (93.5 Avg.)
1½ to 2-in. lumps	86.0	1½ in. slack	96.1, 96.9 (96.5 Avg.)
1 to 1½-in. lumps	90.0	¾ in. slack	98.0, 98.0 (98.0 Avg.)
¾ to 1-in. lumps	93.0		
½ to ¾-in. lumps	94.0		

TABLE III.—DATA FOR TWO NOVA SCOTIA COALS.

LUMPS, IN.	SIZE STABILITY, PER CENT	
	COAL D	COAL E
2 to 3	Two tests..... (75.5 Avg.)	Two tests 76.0, 78.5 (77.0 Avg.)
3 to 4	Two tests..... (70.0 Avg.)	Two tests 71.8, 70.7 (71.0 Avg.)
4 to 6	Three tests 63.9, 62.8, 64.4 (63.5 Avg.)	Three tests 62.0, 67.8 68.6 (66.0 Avg.)
6 to 8	Three tests 49.8 56.0, 51.0 (52.5 Avg.)	Three tests 58.5, 53.5 52.2 (54.5 Avg.)
8 to (12)	Two tests 37.0, 40.1 (38.5 Avg.)	Three tests 36.4, 40.0 40.7 (39. Avg.)

As a rule it has been noted that close checks are not so readily obtained on freshly mined lump coal that has not received much handling, and this applies especially to the larger sizes of the more friable coals. In this connection some results obtained on the larger lump sizes of two freshly mined high volatile bituminous coals from Nova Scotia may be added here (Table III).

At this point attention may be drawn to the "procedure" directions in Section 14 of the supplementary part of the drop shatter test,[3] especially to the last sentence referring to the practice of screening 100-lb. lots of dropped coal in the testing of two 50-lb. samples which reads "This variation in procedure is specially recommended for lumps larger than 8 in., for which size it is advisable that as many as ten lots of approximately 50 lb. each be tested and the average result reported." It is herewith suggested and recommended to fuel technologists using the drop shatter test for lumps larger than the standard 2 to 3-in. size that tests up to five and more be made on each size, and that each test comprise the screening of 100 lb. of dropped coal from two 50-lb. samples, in order to obtain for averaging an ample number of results. An obvious advantage of screening 100-lb. lots of dropped coal is that the weight of each screen size gives the percentage of each size directly and that the calculation of the size stability is thus simplified.

The practice of obtaining the standard 2 to 3-in. size from the larger sized lumps by dropping them in the shatter test machine is an added reason for making a number of drop shatter tests on the larger single sizes. For these tests when conducting the screening on 100 lb. of dropped coal it is necessary to select lumps for two samples that will total 100 lb. rather than making each sample exactly 50 lb.—that is, the sample taken may vary plus or minus, say up to 5 lb., from the stipulated 50-lb. weight. Such a practice is a recommendable means of augmenting the supply of the standard 2 to 3-in. size in cases where the gross sample obtained by screening out this size from the coal to be tested is not sufficient for repeated individual tests to obtain results within the 2 or 3 per cent tolerance allowed. Provided sufficient sample is available, the practice of making as many as five double tests (equal to ten single 50-lb. tests) is applicable to the 2 to 3-in. size as it is to the larger lump sizes.

Comparative Value of Tests on Different Sizes.—The selection of 2 to 3-in. lumps as standard size for the drop shatter test was meant for the purpose of testing and comparing different coals in respect to size stability, while the supplementary method of test for other single and mixed sizes was proposed primarily for testing different sizes of the same coal. Size stability per cent values for different single sizes of the same coal, it has been found when examining the above examples and many others on record, consistently vary directly with the average size of lumps tested. This agrees with the published observances of Smith[8] who selected the 2½ to 3-in. round-hole lumps in his tentative method for Illinois coals but had also tested the 2 to 2½-in. and other sizes. While agreeable to 2 to 3-in. lumps as a standard size he expressed a preference for close sizing and was of the opinion that drop shatter tests on different sizes especially for lumps larger than the standard would not be satisfactory. Yancey[9] in his U. S. Bureau of Mines work on variations of different friability methods as applied to coal tested the ½ to 1-in., 1 to 1½-in., 1½ to 2-in. and the 2½ to 3-in. lumps sized on round-hole screens. A Utah high-volatile bituminous coal, one of the six standard coals used, when tested in duplicate by the four-drop (coke shatter test) method, showed size stability values of 94.0, 90.0, 88.0 and 75.5 per cent for these sizes. Evidently these investigators were interested primarily in selecting the most suitable size of coal as standard for a drop shatter test, whereas the purpose of the A.S.T.M. supplementary method for sizes other than the standard 2 to 3-in. lumps is for determining the relative size stability of different sizes of a given coal.

Such a supplementary testing procedure has merit, since it serves to give the whole picture of the relative stability of the different sized lumps of a coal. In this respect it is specially applicable to freshly mined coal and, therefore, comparative tests may best be conducted at the mine before the coal is handled to any great extent.

Size stability values for the different sized lumps of coals D and E vary from slightly less than 40 per cent for the plus 8-in. lumps to about 75 per cent for the standard 2 to 3-in. lumps and to over 90 per cent for the ¾- to 1-in. and smaller sizes. These smaller sizes, it is to be noted, have values as high as the standard 2 to 3-in. lumps of Pennsylvania anthracite. The two bituminous coals just referred to are to be classed in the "friable" group when examined by the tumbler test. Hence a friable coal as mined may be considered as composed of lumps that will range from low to high size stability when tested by the drop shatter test method. For such friable coals the drop shatter test on the

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[8] C. M. Smith, "An Investigation of the Friability of Different Coals," and "The Friability of Illinois Coals," *Bulletin No. 196* (1929), *Bulletin No. 218* (1930), respectively, University of Illinois Engineering Experiment Station.

[9] H. F. Yancey and R. E. Zane, "Comparison of Methods for Determining the Friability of Coal," *Report of Investigations 3215*, U. S. Bureau of Mines (1933).

different sizes in conjunction with ordinary screen analyses may serve to ascertain to what extent run-of-mine coal will produce coal in the smaller sizes that will have high size stability values and whether it can therefore be shipped and handled without serious size degradation.

Testing of Mixed Sizes.—The supplementary method of test as mentioned above is also applicable for testing mixed sizes, but only certain of its merits will be indicated here. At the Fuel Research Laboratories, a large number of mixed sizes, in addition to different single sizes of the same coal, have been tested

TABLE IV.—SIZE STABILITY AND FRIABILITY VALUES FOR TWENTY-FIVE COALS VARYING WIDELY IN RANK AND FRIABILITY—TOGETHER WITH "SLACK" AND "ABRASION" INDICES.

Size Stability by Drop Shatter Test, per cent		Designation of Coal, Number, Origin, Rank Classification and Grade Symbols	Friability by Tumbler Test, per cent	
On 2- to 3-in. (round hole) Lumps	Slack Index		On 1 to 1½-in. (square mesh) Lumps	Abrasion ("dust") Index
93.5	(3)	1 A—*Pa. Anthracite* (129 - A10 - S1.0 - F28)[a]	19.0	(16)
88.0	(6)	C—*Pa. Westmoreland County* High Volatile A Bituminous	22.5	(14)
85.5	(8)	F—*Welsh Anthracite* (139 - A6 - S1.3 - F22)	30.0	(17)
85.0	(9)	G—*Alta. Mt. Park Area* Medium Volatile bituminous (138 - A10 - S0.7 - F22)	34.0	(20)
82.0	(10)	H—*Alta. Coalspur Area* High Volatile C bituminous (99 - A18 - S0.7 - F22)	31.0	(14)
82.0	(8)	I—*Saskatchewan Lignite* (76 - A6 - S0.7 - F24)	21.0	(7)
		Ohio, Belmont County High Volatile A bituminous		
81.5	(5)	J—(131 - A10 - S5.0 - F20)	28.5	(14)
80.0	(9)	K—(133 - A10 - S5.0 - F20)	27.5	(13)
78.0	(11)	L—(133 - A8 - S5.0 - F20)	27.0	(13)
78.5	(13)	M—*W. Va.—"Pocahontas Lump"* Low Volatile bituminous (138 - A12 - S0.7 - F22)	40.0	(28)
78.5	(6)	N—*Alta. Saunders Area* High Volatile C bituminous (112 - A8 - S0.7 - F22)	40.0	(15)
78.5	(14)	O—*Alta. Coalspur Area* High Volatile C bituminous (103 - A14 - S0.7 - F20)	23.0	(11)
		N. S.—Sydney Area High Volatile A bituminous		
81.5	(10)	4 A—(135 - A10 - S3.0 - F20)	28.0	(10)
77.5	(12)	P—(136 - A6 - S2.0 - F20)	32.0	(15)
77.0	(12)	D—(139 - A6 - S1.6 - F20)	36.0	(16)
76.5	(11)	Q—*Alta. Drumheller Area* Subbituminous B (98 - A8 - S0.7 - F22)	20.0	(8)
		N. S.—Springhill Area High Volatile A bituminous		
76.5	(14)	R—(130 - A12 - S1.6 - F20)	43.0	(24)
75.5	(13)	E—(134 - A10 - S3.0 - F20)	33.0	(19)
75.5	(14)	S—(138 - A10 - S1.3 - F20)	41.5	(24)
70.0	(18)	T—(121 - A12 - S2.0 - F20)	43.5	(20)
		N. B.—Minto Area High Volatile A bituminous		
75.7	(12)	U—(127 - A16 - S5.0 - F20)	36.5	(19)
74.5	(13)	V—(124 - A18 - S5.0 - F20)	38.5	(18)
74.0	(13)	W—*Alta. Prairie Creek Area* High Volatile B bituminous (112 - A16 - S0.7 - F22)	29.5	(13)
68.0	(19)	X—*N. S. Inverness Area* High Volatile C bituminous (112 - A14 - S5.0 - F20)	32.5	(15)
66.0	(19)	7 A—*B. C. Nicola Area* High Volatile B bituminous (111 - A10 - S0.7 - F26)	42.0	(11)

[a] Means B.t.u. 12,900, ash 8.1 to 10 per cent, sulfur 0.8 to 1.0 per cent and softening temperature of ash, above 2800 F. according to symbols adopted in Tentative Specifications for Classification of Coals by Grade (D 389 - 34 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 841 (1934); also 1936 Book of A.S.T.M. Tentative Standards, p. 527.

as part of the physical and chemical survey of coal from Canadian collieries. In these tests practically no difficulty has been experienced in obtaining size stability per cent results on duplicate and triplicate tests agreeing within 2 (and 3) per cent, when restricting the largest size lumps in such mixed sizes to less than 4 in. For best results, however, strict attention must be paid to the directions given in Section 13 under "Preparation of Sample" of the drop shatter test[3] reading as follows:

> The sample of coal for test shall be prepared in accordance with Sections 5 and 6. For slack coals and mixed sizes the sample shall be carefully prepared either by the process of quartering or by reassembling the different sizes in the proportion obtained in the preliminary screening of the lot of coal to be tested. For ¾-in. and smaller size slack coals, quartering is satisfactory, while for larger size slack coals and for blends of two or more single sizes the latter reassembling method is recommended. Before dropping, the sample shall be rescreened on the same series of screens selected for screening the dropped coal.

Drop shatter tests on a composite run-of-mine coal, through a 4-in. screen for example, in comparison with tests on the 2- to 3-in. and smaller single sizes serve to demonstrate their comparative handling properties. Referring to the data given above on coal 4A it will be noted that, whereas the size stability of the 2 to 3-in. lumps was 83.5 per cent, the composite mixed 4-in. coal was found to be 93.5 per cent, midway between the stabilities of the smaller ¾ to 1-in. and ½ to ¾-in. sizes. This indicates the cushioning effect of the smalls and fines on the larger lumps in the 4-in. coal, which lumps, in this case, amounted to nearly 40 per cent larger than 2 in. Such results support the viewpoint that the best way to ship friable coals in order to prevent size degradation is as run-of-mine or slack, and when the market calls for sized coal the sizing should be done as near as possible to the point of delivery to the ultimate consumer.

Slack Index.—As described in explanatory note 2 at the end of the drop shatter test,[3] "the percentage passing the screen with the ¾-in. openings may be reported to the nearest whole per cent as the 'slack index' to indicate the comparative slack-producing characteristics of the particular size of coal tested." Coals C and D as below are good examples of the relation of the slack indices to size stability per cent values on different coals, as well as on different sizes of each coal:

	Coal C		Coal D	
	Size Stability, per cent	Slack Index	Size Stability, per cent	Slack Index
2 to 3-in. lumps.	88	6	77	12
3 to 4-in. lumps.	84	6	71	13
4 to 6-in. lumps.	79	5	66	12
6 to 8-in. lumps.	..	.	51	14

These results illustrate that, corresponding to a difference of ten in size stability, the slack index of the more friable coal was double that of the more stable coal, but that this index does not change materially and progressively for the lumps larger in size than 2 in. The reporting of the slack index as supplementary information on a given coal may advisably, therefore, be restricted to the standard 2 to 3-in. size.

GENERAL COMPARISONS AND CONSIDERATIONS

A comparison of the values obtained by the tumbler and drop shatter test methods for a series of twenty-five coals varying widely in friability is shown in Table IV. This table comprises both the size stability and the friability values determined according to the two drop shatter test and the 1-hr. tumbler test standardized procedures. Its contents deserve detailed study and comment, but only some of the more salient points will be discussed here.

The coals are listed according to the descending order of their size stability per cent values for standard 2 to 3-in. size lumps. The number of coals shown is too limited to draw other than very general comparisons and conclusions. Generally the corresponding order of the friability per cent values is in the reverse order—from low to high—but considerable irregularity and overlapping is to be noted. This is illustrated by the following comparison of groupings (where SSP means size stability per cent):

Size Stability, per cent, Groups and Values	Corresponding Friability, per cent, Groups and Values
High SSP group	Low friability group
Above 90	Below 20
Medium SSP group	Medium friable and friable groups
Sub-group A, 80 to 90	20 to 35
Sub-group B, 70 to 80	20 to 45
Low SSP group	Medium and friable groups
Below 70	30 to 50

Similarly, if the twenty-five coals are arranged in order of their friability per cent, as obtained by the tumbler test, it will be found that the non-friable group with friability per cent values below 20 contains the coal with SSP above 90, that the medium friable group comprising sub-divisions A (20 to 30) and B (30 to 40) contain coals with SSP values from 88 to 68 and that the "friable" (40 to 50) group contains coals with SSP values ranging from 78 to 66.

Consideration of such a series of coals according to their descending size stability values is to be preferred because the SSP values are believed to be more in accordance with the size degradation taking place in the ordinary commercial handling of lump coal. Incidentally those producers and distributors of coal marketing specially sized products where stability of size is important would no doubt prefer to have their coal designated in terms of "size stability" as adopted for the drop shatter test rather than in terms of "friability" as adopted in the tumbler test. On the other hand those marketing coals for use such as pulverized fuel, by-product oven coking, etc., where stability of size is not an important factor, may welcome having their coal designated in terms of friability, providing the corresponding size stability factor was sufficiently high to meet storage and other requirements.

Abbreviations for Expressing Handling Properties of Coal:

In accordance with these viewpoints it is advisable to determine and report both the size stability and friability values. This may be done by stating these values for a given coal verbatim or by the use of an abbreviation as per the following example:

Handling properties.................. 88–22.

Here 88 would be the size stability per cent, to the nearest whole number, as obtained by the drop shatter test on standard 2 to 3-in. lumps, and 22 would be the friability per cent, also to the nearest whole number, as obtained by the tumbler test method. If symbols are preferred as prefixes the expression *Ss 88 – Fr 22* could be used where "Ss" and "Fr" would mean size stability per cent and friability per cent respectively. Such a two-fold expression would serve to differentiate between two or more coals showing approximately the same size stability values but having different friability per cent values, and *vice versa*.

If and when considered desirable to report the grindability, in conjunction with the size stability and friability, either by the ball-mill method, or by the Hardgrove-machine method, this may be done by adding a grindability index to the friability values in either abbreviated or symbol form.

History and Screen Analysis of Coal:

It has been observed that two shipments of coal from the same mine and the same seam sometimes show appreciably different size stability per cent values by the drop shatter test and this applies also to the tumbler test. Such a difference is as a rule explainable either by a variation in the coal seam or by a variation in the amount of handling before testing. If, for example, the 2 to 3-in. lumps obtained as sample for the drop shatter test are derived from the larger, more resistant lumps of screened coal after a great deal of handling, the size stability per cent result may be noticeably higher than when the sample for testing is screened out of the smaller size run-of-mine product, in which the less resistant lumps have been protected from breakage by smalls and fines. In this respect, therefore, it is advisable to know and record, if available, both the history of the lot of coal to be tested and its screen analysis prior to testing.

GENERAL SERVICEABILITY AND CONCLUSIONS

A casual survey of the contents of Table IV demonstrates the closeness to each other of the results reported. It is to be noted that, among the size stability and friability per cent results reported to the nearest 0.5 per cent, as many as seven coals show SSP (size stability per cent) values within 2, the tolerance tentatively adopted for two or more tests on the same coal, and that each of two sets of four coals have friability per cent values within this limit. This, therefore, means that the tests are serviceable for ascertaining the similarity of coals in respect to size stability and friability, rather than for determining values within narrow limits in order to emphasize their dissimilarity.

In conclusion, the general serviceability of the drop shatter and tumbler test methods may be stated to be a means of placing coals in groups in respect to size stability per cent and friability per cent; a range of ten has been suggested for individual groups. The testing of coals by the two methods is advisable in order to indicate the relative handling properties in respect to friability. The drop shatter test serves for determining the resistance to breakage due to a lesser amount of handling as in the ordinary preparation and transportation of lump coal, while the tumbler test serves for determining the liability to further breakage and ease of breakage when the coal is subjected to rougher handling as in mechanical conveyors, feed devices, crushers, etc.

DISCUSSION

MR. J. A. TAYLOR[1] (*presented in written form*).—Tests made with the small jar tumbler show that the 1-hr. test is just as duplicable as the former 3-hr. test. This should uphold the 1-hr. test as tentatively adopted and eliminate the 3-hr. test.

An attempt is made to show the effect of closer sizing in selecting the sample. The conclusion reached is that the 1.5 to 1.05-in. sample should be fairly uniform with respect to the sizes in between. Relatively large errors may be made in extreme cases, but, in general, any error from this source will be offset by the error in the determination.

Regarding values obtained with the drop shatter test, three methods of selecting samples showed little difference in final results with the 2 to 3-in. size: namely, from run-of-mine in storage bin, from shatter tests made with larger sizes, or from lumps broken down with the hammer. The over-all 2 per cent accuracy of the test undoubtedly covers any errors from this type of sampling.

Many tests using various sized coals in the drop shatter test are shown and discussed. The principal value in testing large sizes seems to be that a considerable amount of the so-called standard 2 to 3-in. size can be produced for use in a standard test. On large sizes the checks are poor and numerous tests have to be made to obtain a good average. In testing the small sizes the main point is that size stability per cent is large even with quite friable coals and that there is, therefore, little distinction between different coals.

The testing of mixed coals by the drop shatter test seems to me to afford very little information other than that which is already obvious without the test. The size stability index resulting from a test on a run-of-mine coal with a large average size cannot compare with the size stability index from a test with a run-of-mine coal having a small average size. The advantage pointed out for such testing seems irrelevant.

One of the main features of this paper is the classification of coals both by the friability per cent from the jar tumbler test and by the size stability per cent from the drop shatter test. The authors arbitrarily set up various groupings which cover all coals over a very large range. The coals do not fall into exactly the same groupings with both tests as tentatively set up by the authors, but on the whole there is some agreement. In concluding, the authors recommend that testing be done by both methods. If some sort of average between the friability per cent and size stability per cent could be arranged, these tests might be useful for classifying coals, but of course it must be realized that all testing of this nature is highly empirical and not very fundamental.

MR. MARTIN FRISCH.[2]—I know that Mr. Gilmore has done a great deal of work on the study of friability and

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[1] Research Assistant in Fuel Technology, Mineral Industries Experiment Station, The Pennsylvania State College, State College, Pa.

[2] Chief Engineer, Boiler and Pulverizer Division, Foster Wheeler Corp., New York City.

"grindability," and I wish to inquire whether he has been able to derive a correlation between friability and "grindability." Our experience indicates that there may not really be much difference between the usefulness of friability and "grindability" tests in indicating the probable behavior of a coal when undergoing rough handling, whether in shipment or in a crusher or in a pulverizer. As a matter of fact, when I first became interested in the pulverizing characteristics of coals, before there was such a thing as a "grindability" test, I found it possible to correlate in a rough manner many of the friabilities reported by Mr. Cloyde Smith[2a] of the University of Illinois, with actual performances in pulverizers. It is quite possible, now that results of carefully made friability tests can be checked with an accuracy of 2 per cent, that a correlation could be obtained between friability and "grindability," and a great deal of the work done to measure friability could be made available to users and makers of pulverizing equipment.

It may be that results of friability tests will prove to be just as useful as the results of "grindability" tests. If that should prove to be the case, the necessity for the "grindability" test may disappear. It certainly would be an advantage if coal shippers as well as coal users could use the same test and index: namely, the friability test as a basis for estimating the ease of degradation of coal whether in handling, shipping, crushing or pulverizing.

The writer's work and experience as described in our paper on grindability clearly indicates that "grindability" is an arbitrary and not an absolute index of a coal and that the relative ranking of a material as to its ease of degradation depends on the kind of test used to determine the "grindability." Each method of ranking assigns a different relative rank to a coal and that rank is generally different from the ranking assigned by actual pulverizer tests.

MR. R. D. HALL.[3]—This paper is extremely practical. There are two things that we need to discover. We are trying, on the one hand, for classification of coal as coal, and in addition we are trying to find out the characteristics of coal as mined and delivered. This paper has to do with coal as mined and delivered. Now, coal in the bed is doubtless very much stronger than the coal as mined and delivered, because it has not been shattered by the explosions by which the coal is brought down.

Some time ago J. F. Joy told me that his machine for cutting coal out of the bed without shooting but with the aid of a hydraulic mat which separates the blocks, is not successful in bringing down big blocks of coal until the advance of the machine has exceeded three cuts ahead, of former mining, that is, 18 ft., showing that in those 18 ft. there was a certain degree of shattering; so you can see that when we go into the mine we really never see the coal as it is but we rather see the coal after it has been shattered. The original coal, therefore, has much greater strength than the form in which it is tested. If it were taken out of the solid and tested it would be found to be much stronger.

The harder the coal, the more we shoot it, and, therefore, the more we crevice it. Consequently, the harder the coal, the more damage we do to it and the less we show its real and true strength—a consideration that we should keep in mind. The observations made

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[2a] Cloyde Smith, "An Investigation of Friability of Different Coals," *Bulletin 196*; "The Friability of Illinois Coals," *Bulletin 218*, Engineering Experiment Station, University of Illinois.

[3] Engineering Editor, *Coal Age*, New York City.

by Messrs. Gilmore and Nicolls are on mined and delivered coal and not coal as it is in the bed. I have sometimes questioned whether the studies which have been made into the plasticity of coal by the U. S. Bureau of Mines should not be to some extent discounted because the explosives have been used in driving the roadways in which those tests are being made, for the coal is probably creviced to some extent and they did not have coal in its pristine condition. On the other hand, when the Bureau of Mines made its experiments on the compression of coal in blocks they mined that coal out very carefully. They did not shoot it, and the result is they probably found that they had a stronger coal to deal with, and that may have had some effect on the relation between the two classes of investigation. It may prove that after all there is some value in those block coal investigations which has not been duly considered. In the one case they have blocks which have no side support; in the other case they have coal which, while it had a side support and rear support, is at the same time already more or less creviced by mining. Now, what are we after? Are we after the classification of the coal as it is in the ground or are we after the classification of coal as it is prepared for the market or at the point of delivery?

MR. R. E. GILMORE.[4]—Replying to Mr. Frisch I may say that we have at Ottawa grindability and friability data on a number of coals. The results show a general relation only, between friability and grindability—the more friable coals, of course, having higher grindability indices. The friability method employing 1 to $1\frac{1}{2}$-in. (square mesh) lumps serves for indicating the inherent weakness of the coal when subjected to handling such as in mechanical feed devices and crushers, whereas grindability tests are desired for indicating relative power requirements for reducing the coal particles to the fineness required for pulverized fuel combustion. Hence friability tests will not likely do away with the necessity of making supplementary grindability tests since the latter afford a means of differentiating between coals having approximately the same friability per cent values.

Replying to Mr. Hall I might emphasize that the drop shatter (size stability) test is considered to be serviceable for testing the relative strength of different sized lumps from the time the coal is mined to when it reaches the consumer, as well as for comparing the standard 2 to 3-in. sized lumps, and other sizes for that matter, of different coals.

MR. L. A. SHIPMAN[5] (*presented in written form*).—In connection with the drop shatter tests, I wish to offer some information which was obtained in breaking strength tests on coal. The results of these tests indicated that with some coals the difference between the breaking strength with and against the grain is so great that the results from a drop shatter test with these coals could hardly be compared with the results on a practically grainless coke or a coal in which practically no difference is obtained with and against the grain. In the case of some coals where there are two or more strata in the seam having different breaking strengths either with or against the grain, the results of a drop shatter test would be influenced by the percentages of the different strata included in the drop test.

In applying breaking strength tests to coal samples, we developed some interesting facts. In these tests, after

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[5] Combustion Engineer, Southern Coal and Coke Co., Knoxville, Tenn.

facing the sides of the coal to be tested with plaster of Paris to insure smooth faces for applying the load, the measured pieces of coal were placed in the machine and the load applied. In testing coal from the Harlan seam, six samples of two kinds of coal were taken and were prepared so as to test three samples with the grain and three samples against the grain. Load was applied until the coal broke and would not pick up the weight of the scales again.

SIZE OF BLOCK, IN.	PRESSURE APPLIED	BREAKING STRENGTH, LB.	STRENGTH, LB. PER SQ. IN.
No. 1.—Coal with Splint Streak:			
6½ by 6¼ by 6¼ high	With grain	91 600	2255
5½ by 7 by 6 high	Against grain	162 000	4155
No. 2.—Black Shiny Coal with Little Apparent Grain:			
5½ by 6½ by 6 high	With grain	112 900	3158
6½ by 7½ by 6½ high	Against grain	137 400	2818

The point I wish to bring out is that in some coals there is a large difference in the breaking strength with the grain as against the grain.

I have an instance of one coal having a strength of 1477 lb. per sq. in. with the grain and 5020 lb. per sq. in. against the grain. The drop shatter test with a coal having such a large difference between the breaking tests will show a wide variation in results depending on the number of pieces dropping with and against the grain.

In the case of a coal with practically no grain, where the breaking strength both with and against the grain is nearly the same, fairly close results should be obtained where the sizes or weights of pieces are fairly uniform.

One other item of interest is that where the pieces of coal had broken down in this test, a fairly large block of coal remained which could be handled fairly well but which could be pulled apart with some effort. When coal is taken from mine pillars where the coal has taken weight to the point of breaking down, a considerable number of medium and large pieces will carry over the screen and be loaded in the block or egg car. In such cases, where the coal is already fractured internally, the drop test would not check closely with another sample which contained none or a smaller percentage of the fractured coal.

MR. R. J. HOLDEN[6] (*by letter*).—For several years the Engineering Experiment Station of the Virginia Polytechnic Institute has been making coal-friability tests. The primary purpose of these tests was to determine what relation, if any, exists between coal friability and the geological experiences through which the coal has gone. The secondary purpose was the matter of friability of coal of various types without regard to geological relations.

In our earlier testing work we used porcelain cylinders provided with baffle plates. For certain coals this gave satisfactory results, but on other coals a large part of the off-fall represented abrasion and not shattering. For our main purpose, it was essential to have a method which would apply to all coals regardless of rank and one in which the shattering factor was more prominent. Tests were considered which theoretically would reduce the abrasion factor to zero, but these were discarded in favor of a method which would include what seemed to be an appropriate abrasion factor.

The Deval type abrasion rattler, which is a standard machine used for testing the resistance to abrasion of stone used for road construction, was adopted. This machine has cylinders 20 cm. (approximately 8 in.) in diameter by 34 cm. (approximately 13½ in.) long. These are set with the cylinder axis

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[6] Professor of Geology, Virginia Polytechnic Institute, Blacksburg, Va.

at an angle of 30 deg. with the shaft, which revolves at 30 r.p.m. The material in the cylinders is then dumped back and forth between the ends of the cylinders as the shaft rotates.

Trials were made to determine the most satisfactory lengths of time for tests, the fines were removed at 15-min. intervals, and time *versus* off-fall curves plotted through 3-hr. periods. It was found that for different coals there was gradual divergence in the curve through 2 hr., but thereafter the curves were essentially parallel. The 2-hr. period was, therefore, adopted as the standard time. The charge used was 3000 g.

For our purpose it was essential to have a unit friability figure and it was desirable to obtain this directly from the tests rather than by computation from the different sizes of the off-fall.

It was found by experience that in general when coals broke up on friability tests they broke to fairly small particles. Accordingly, the No. 4 sieve was adopted as standard, and the percentage of material passing this sieve was adopted as the unit friability figure. This method gave us a wider range of friability than had been obtained by methods which had been used previously.

Different types of coal tested gave friabilities as follows: Cannel (one sample) 2.4, anthracite 8 to 11, hard block coals 14 to 20, semi-anthracite 20 to 25, medium granulated 20 to 40, high granulated 40 to 60.

As a standard method for friability this gives a wide range of results, is applicable to all kinds of coal, uses standard apparatus, and gives a result directly from the experimental data. If a dust factor is desirable, it can be obtained by subsequent screening of the material passing the No. 4 sieve.

PULVERIZER PERFORMANCE AS AFFECTED BY GRINDABILITY OF COAL AND OTHER FACTORS

By Martin Frisch[1] and A. C. Foster[1]

The Society after a cooperative investigation of four methods has recently published two as tentative methods of test for rating coals as to their relative pulverizing characteristics, now commonly termed grindability.[2] Makers and users, alike, of pulverizing equipment are interested in the meaning of these ratings in terms of actual performances of commercial pulverizers. Data are herewith presented to show the relation between results of various laboratory methods for determining grindability and between grindability and commercial pulverizer performance.

The Society became interested in grindability shortly after Cross[3] in 1926 first developed and described a laboratory test procedure for ranking coals in accordance with index numbers proportional to the amount of new surface produced per unit weight of sample during a prescribed laboratory treatment in a standard laboratory jar mill. Since then others have investigated the problem and advanced its understanding.

Hardgrove[4] in 1931 described a test procedure, now known as the Hardgrove-machine method, which he developed for ranking coals in accordance with ratios of the new surface produced during a laboratory test of a coal and the new surface produced when testing an arbitrarily selected standard coal in a like manner. Like Cross, Hardgrove used surface estimates for ranking coals, but he employed, instead of the standard jar laboratory mill, a treating machine of special design.

Frisch and Holder[5] in 1933 presented results of a simplification of Hardgrove's method, hereafter referred to as the F. W. method, which they had developed and used since 1931. By this method, coals are ranked in accordance with direct fineness measurements of the Hardgrove machine test product as given by the amount passing some one sieve such as the No. 300, No. 200, or No. 100 sieve, eliminating the necessity of calculating surface ratios from the results of screen analyses involving the use of seven sieves and seven surface factor estimates.

Baltzer and Hudson[6] in 1933 described a new procedure known as the F. R. L. method, employing the standard laboratory jar mill. The grindability is the amount of minus 100-mesh material produced in the last of three equal prescribed cycles. In this method an effort to simulate actual pulverizer operation

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[1] Chief Engineer, and Pulverizer Engineer, respectively, Boiler and Pulverizer Division, Foster Wheeler Corp., New York City.

[2] Tentative Methods of Test for Grindability of Coal by the Ball-Mill Method (D 408 - 35 T), and for Grindability of Coal by the Hardgrove-Machine Method (D 409 - 35 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, pp. 854 and 857, respectively (1935); also 1936 Book of A.S.T.M. Tentative Standards, pp. 532 and 535, respectively.

[3] Progress Report on Pulverizing Characteristics of Coal, A.S.T.M. Committee D-5, Subcommittee VII. Private communication by B. J. Cross (1927).

[4] R. M. Hardgrove, "Grindability of Coal," *Transactions*, Am. Soc. Mechanical Engrs., Fuels and Steam Power Division, Vol. 54, p. 37 (1932).

[5] Martin Frisch and G. C. Holder, "Correlation of Grindability with Actual Pulverizer Performance," *Combustion*, Vol. 4, No. 12, June, 1933, p. 29; Vol. 5, No. 1, July, 1933, p. 34.

[6] C. E. Baltzer and H. P. Hudson, "Fuel Research Laboratories Simplified Method for Rating the Grindability or Pulverizability of Coal, Correlated with Van Brunt—Cross and Hardgrove Methods," Canada Department of Mines, Division of Fuels and Fuel Testing, Ottawa, February 1, 1933.

is made by removing the finished product at the end of each cycle and replacing it with an equal amount of original feed. Were this carried out for a sufficiently large number of very short cycles, the mode of operation of a pulverizer with continuous feed and discharge would be approximated better than by any other method so far proposed.

Yancey, Furse, and Blackburn[7] in 1934 described a method, now known as the Bureau of Mines ball-mill method, employing a special ball mill of standardized dimensions. By this method coals are ranked in accordance with the reciprocal of the number of revolutions required to pulverize a 500-g. sample in eight equal steps or cycles until 80 per cent is removed as minus 200-mesh material.

In 1934 the Society's Committee D-5 on Coal and Coke undertook a cooperative investigation to compare the then best-known procedures in order to determine their relative accuracy, reproducibility, speed, and equivalence in ranking the materials tested. These were the Cross, Hardgrove, U. S. Bureau of Mines and the F. R. L. (Canada Department of Mines) methods. The results of this cooperative investigation in which participated the U. S. Bureau of Mines, Babcock & Wilcox Co., Canada Department of Mines, Fuel Engineering Co., and Foster Wheeler Corp., hereafter referred to as laboratories A, B, C, D, and E, respectively, were summarized, analyzed, and discussed in 1936 by W. A. Selvig.[8]

As a result of the cooperative investigation the Society adopted the Bureau of Mines ball-mill and the Hardgrove-machine methods as tentative methods of test (D 408 - 35 T and D 409 - 35 T, respectively).[2] Since their approval the Subcommittee on Pulverizing Characteristics of Coal of the Society's Committee D-5 on Coal and Coke has voted to revise Tentative Method D 409 - 35 T to specify the use of the fineness of the Hardgrove machine product as measured by the amount passing the No. 200 sieve as the basis for calculating grindability from the empirical equation:

$$\text{Grindability} = G = 13 + 6.93\,W$$

where W is the weight in grams of the 50-g. sample passing the No. 200 sieve.

Meanwhile Sloman and Barnhart[9] in 1935 described a very simple method developed by them at the Carnegie Institute of Technology for rating coals as to grindability in accordance with surface calculations. This method, known as the C.I.T. roll test, involves the crushing of a small sample by passing a standard roller over it 10 times.

Yancey and Geer[10] in 1935 reported the results of further investigations of procedures for determining grindability including comparative tests of the C.I.T., Hardgrove, and ball-mill methods.

Black[11] in 1936 described grindability tests with the ball-mill method based on the use of samples of the same volume instead of the same weight for each test.

After the adoption of the tentative methods, a study was undertaken by the authors to determine the relation between the F.W. coal ratings used by them since 1931, and other grindability ratings so as to facilitate the correlation of the results of commercial pulverizer tests

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[7] H. F. Yancey, O. L. Furse and R. E. Blackburn, "Estimation of the Grindability of Coal," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 108, pp. 267-283 (1934).

[8] W. A. Selvig, "Check Determinations of Grindability of Coal by Various Methods," *Report of Investigations 3301*, U. S Bureau of Mines, February, 1936.

[9] Harold J. Sloman and Arthur C. Barnhart, "The Relative Grindability of Coal," *Transactions*, Am. Soc Mechanical Engrs., Fuels and Steam Power Division, Vol 56, pp. 773-779 (1934).

[10] H. F. Yancey and M. R. Geer, "Further Investigation of Methods for Estimating the Grindability of Coal," *Contribution No. 94*, New York Meeting, Am. Inst. Mining and Metallurgical Engrs., February 1936.

[11] C. G. Black, "Investigation of Procedure for Determination of Coal Grindability by the Ball-Mill Method," *Contribution No. 95*, New York Meeting, Am. Inst. Mining and Metallurgical Engrs., February, 1936.

TABLE I.—RESULTS OF GRINDABILITY DETERMINATIONS OF FIVE SELECTED COALS BY VARIOUS METHODS.

Basis	Coal	Laboratory A	Laboratory B	Laboratory C	Laboratory D	Laboratory E	Laboratory C.I.T.	Average	Laboratory E F. W. Method	
BALL-MILL METHOD										
Revolutions for 80 per cent Passing No. 200 Sieve	No. 1	595	685	603	624	......	......	627		
	No. 2	1 057	1 119	1 085	1 047	......	......	1 077		
	No. 3	1 166	1 267	1 217	1 226	......	......	1 219		
	No. 4	1 342	1 360	1 345	1 329	......	......	1 344		
	No. 5	1 939	1 940	1 968	2 019	......	......	1 967		
Grindability, lb. per hr.	No. 1	4.45	3.86	4.39	4.24	......	......	4.25		
	No. 2	2.50	2.36	2.44	2.53	......	......	2.42		
	No. 3	2.27	2.09	2.17	2.16	......	......	2.20		
	No. 4	1.97	1.94	1.97	1.99	......	......	1.95		
	No. 5	1.36	1.36	1.34	1.31	......	......	1.34		
HARDGROVE-MACHINE METHOD										
f100, Percentage Passing No. 100 Sieve	Jenner	......	......	......	......	......	......	......	39.3	
	Emore	40.0	38.7	39.4	......	......	......	39.4	39.5	
	No. 1	40.1	39.4	40.6	......	......	......	40.0	35.4	
	No. 2	27.0	26.7	27.0	......	......	......	26.9	20.4	
	No. 3	25.1	23.0	23.0	......	......	......	23.7	18.2	
	No. 4	21.2	20.1	21.4	......	......	......	20.9	16.4	
	No. 5	13.5	13.0	13.7	......	......	......	13.4	10.0	
f200, Percentage Passing No. 200 Sieve	Jenner	......	......	......	......	......	......	......	25.5	
	Emore	25.6	24.1	26.2	......	......	......	25.5	24.3	
	No. 1	25.5	24.7	27.1	......	......	......	25.8	23.8	
	No. 2	15.7	15.3	15.9	......	......	......	15.6	12.7	
	No. 3	13.5	14.0	13.2	......	......	......	13.6	11.2	
	No. 4	10.4	9.5	11.3	......	......	......	10.4	9.0	
	No. 5	6.7	6.8	7.2	......	......	......	6.9	5.4	
f300, Percentage Passing No. 300 Sieve	Jenner	......	......	......	......	......	......	......	24.4	
	Emore	21.3	19.0	20.4	......	......	......	20.2	15.9	
	No. 1	21.2	19.9	21.8	......	......	......	20.9	17.3	
	No. 2	13.1	10.9	12.4	......	......	......	12.1	9.3	
	No. 3	11.0	10.6	10.3	......	......	......	10.6	8.7	
	No. 4	8.3	7.1	8.4	......	......	......	7.9	6.2	
	No. 5	5.5	4.8	5.7	......	......	......	5.3	4.0	
Surface Units, S	Jenner	......	......	......	......	......	......	......	29 485	
	Emore	27 692	25 900	27 630	......	......	......	27 074	24 586	
	No. 1	28 180	26 745	28 390	......	......	......	27 771	24 403	
	No. 2	18 500	17 105	18 236	......	......	......	17 947	14 234	
	No. 3	16 590	15 949	15 754	......	......	......	16 097	12 523	
	No. 4	14 030	12 814	14 417	......	......	......	13 754	11 086	
	No. 5	10 120	9 489	10 462	......	......	......	10 024	7 784	
Surface Ratios, Grindability, $G = 100 \frac{S_n}{S_e}$ where e = Emore	Jenner	......	......	......	......	......	......	......	120.0[a]	100.0[b]
	Emore	100.0	100.0	100.0	......	......	......	100.0	100.0	83.4
	No. 1	100.5	103.4	102.8	......	......	......	102.2	99.4	82.6
	No. 2	66.9	66.5	66.0	......	......	......	66.5	57.9	48.2
	No. 3	60.1	61.6	57.1	......	......	......	59.6	53.1	44.1
	No. 4	50.9	49.5	52.2	......	......	......	50.9	45.2	37.5
	No. 5	36.6	36.6	37.9	......	......	......	37.0	31.8	26.4
CANADA DEPARTMENT OF MINES F.R.L.										
Minus 100 Mesh Material Discarded, g.	No. 1	321	308	372	364	312	......	335		
	No. 2	205	191	236	237	186	......	211		
	No. 3	207	187	221	221	183	......	205		
	No. 4	207	202	212	127	145	......	175		
	No. 5	127	41	42	9.6	7.0	......	45		
CROSS										
Surface Index	No. 1	1036	886	933	542	582	......	796		
	No. 2	526	519	401	262	293	......	400		
	No. 3	469	519	174	37	185	......	277		
	No. 4	326	251	111	13	49	......	150		
	No. 5	137	125	44	4.9	5	......	63		
C.I.T. ROLL TEST										
Surface Ratios, $G = 100 \frac{S_n}{S_1}$	No. 1	100	......	......	......	......	100.0	100.0		
	No. 2	64	......	......	......	......	58.5	61.0		
	No. 3	63	......	......	......	......	47.6	56.0		
	No. 4	48	......	......	......	......	42.0	45.0		
	No. 5	43	......	......	......	......	31.6	37.0		

[a] Standard coal rating 100 = Emore.
[b] Standard coal rating 100 = Jenner.

and day to day pulverizer performance records known to them, with these other grindability ratings. The methods studied were: the four investigated during the cooperative investigation; the F.W. method used for ranking the coals on which most of the commercial pulverizer performance data hereafter discussed were obtained; the C.I.T. roll test; and the constant volume modifications of the two tentative methods.

During the A.S.T.M. cooperative grindability tests, laboratory E treated each of the five coals investigated also by the F.W. method and thereby established the convertibility of results obtained by this method into results obtained by the ball-mill method (D 408 – 35 T) and the Hardgrove-machine method (D 409 – 35 T). This made it possible for the authors to derive from results of parallel tests of the same five coals by five laboratories a satisfactory correlation between F.W. grindability index numbers and grindabilities obtained by other methods and laboratories. A large amount of actual commercial pulverizer performance data obtained on coals which had been tested and rated by the F.W. method only and had been previously correlated by the authors only in terms of F.W. index numbers is now correlated with grindabilities obtained by other methods and laboratories.

TABLE II.—RELATIVE RATINGS OF COALS BY VARIOUS LABORATORY METHODS.

NOTE.—For significance of symbols see Fig. 4.

Coal	Relative Ratings Based on Laboratory Tests—Coal No. 1 = 100														F.W. Index Number, Percentage passing No. 200 sieve
	Ball-Mill Method		Hardgrove-Machine Method				F.W. Method Basis				F.R.L.	Cross	C.I.T.		
	Original Standard Basis, D 408 – 35 T, 100 W_n/W_1	Adjusted as in Fig. 6, 100 W_n/W_1	Surface D 409 – 35 T, 100 S_n/S_1	Passing No. 100 Sieve, per cent, 100 a_n/a_1	Passing No. 200 Sieve, per cent, 100 b_n/b_1	Passing No. 300 Sieve, per cent, 100 c_n/c_1	Surface, 100 S_n/S_1	Passing No. 100 Sieve, per cent, 100 a_n/a_1	Passing No. 200 Sieve, per cent, 100 b_n/b_1	Passing No. 300 Sieve, per cent, 100 c_n/c_1	Weight Passing No. 100 Sieve, 100 a_n/a_1	Surface, 100 S_n/S_1	Average Laboratory A and C.I.T., 100 S_n/S_1		
No. 1	100	100	100	100	100	100	100	100	100	100	100	100	100		23.8
No. 2	57	56	65	67	58	58	58	58	53	54	63	50	61		12.7
No. 3	52	50	58	59	51	51	53	51	47	50	61	35	56		11.2
No. 4	46	44	50	52	39	38	46	46	38	36	53	19	45		9.0
No. 5	32	30	36	34	26	25	32	28	23	23	13	8	37		5.4
Emore	......	......	100	99	99	97	101	111	102	92	......	......	......		24.3
Jenner	......	......	......	......	......	......	121	111	107	141	......	......	......		25.5

RELATIVE GRINDABILITY RATINGS FROM LABORATORY TESTS

In Tables I and II are summarized the average results and relative ratings obtained.

Hardgrove Method:

The results of the Hardgrove-machine method are herewith expressed, not only in the usual surface ratios, but also in surface units, and as numbers representing the percentages passing the Nos. 100, 200 and 300 sieves, respectively.

For calculating surface ratios, laboratories A, B, and C used Emore coal rated 100 as the standard. Laboratory E used Jenner coal, its own standard rated 100, because its tests of the Emore coal were not completed in time for inclusion in its report. Subsequently, laboratory E did test Emore coal, and in Table I its surface ratios are expressed in terms of Emore as well as Jenner coals.

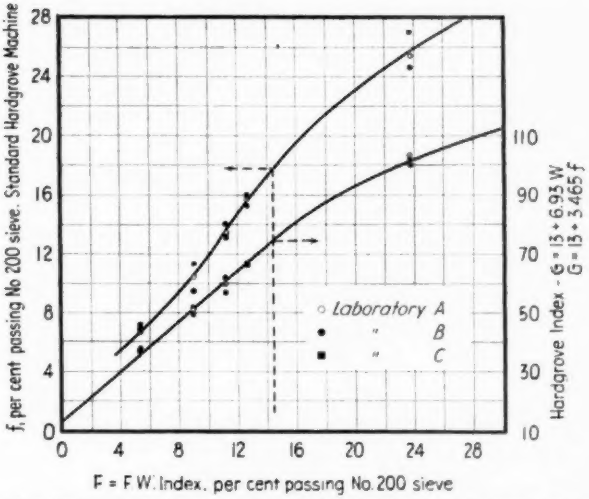

FIG. 1.—Relation Between F. W. Index and Grindability by Hardgrove-Machine Method (D 409 – 35 T).

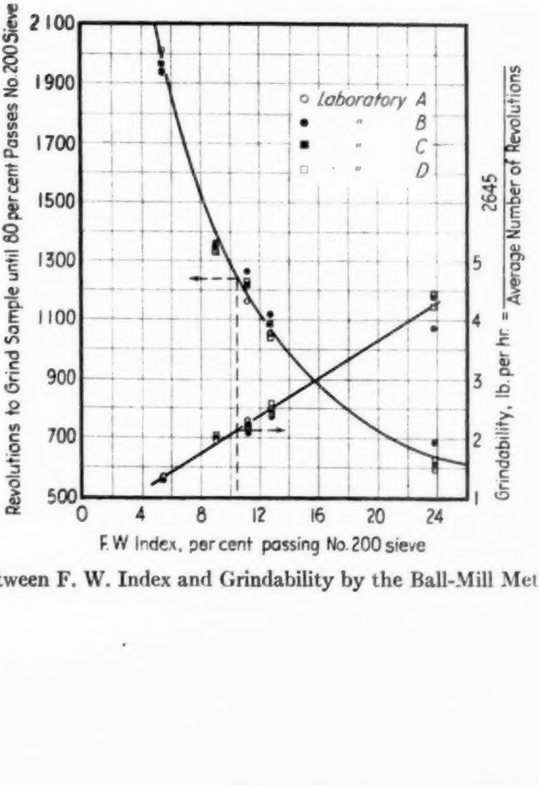

FIG. 2.—Relation Between F. W. Index and Grindability by the Ball-Mill Method (D 408 – 35 T).

Laboratories A, B, and C used standard Hardgrove testing machines. These were run at a speed of 21 r.p.m. for a total of 60 revolutions. Laboratory E used a machine of the same type and general design but built in its own shops in 1931 from cuts appearing in Hardgrove's original paper.[4] Early tests indicated that better consistency could be obtained with this particular machine when operated for a total of 75 revolutions than for the prescribed 60 revolutions. Therefore, since hundreds of samples had been previously tested in this manner for correlation with actual pulverizer performances, it was deemed inadvisable to change to 60 revolutions, recalibrate the machine and establish a new correlation, and the five samples supplied by the committee were tested for 75 instead of 60 revolutions. The machine had been standardized to produce approximately 29,500 to 30,000 new surface units on Jenner coal, arbitrarily selected in 1931 as one standard coal for rating pulverizers. F.W. index numbers and surface ratios, therefore, obtained by laboratory E during the cooperative investigation and corresponding indices obtained by the other laboratories by the Hardgrove method are not exactly the same, but they are inter-convertible by using the correlating curves on Fig. 1. Surface ratios for the hardest coals tested on the F.W. machine referred to Emore coal are approximately 90 per cent of those obtained with standard Hardgrove machines, while for the softest coals they are almost the same.

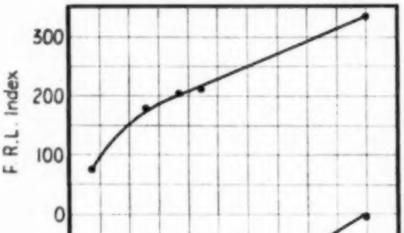

FIG. 3.—Approximate Relation Between F. W. Index and Grindability by F. R. L. and Cross Methods.

Ball-Mill Method:

Correlations between F.W. index numbers and grindability ratings derived by the ball-mill method are shown on Fig. 2 on which (*a*) the number of revolutions required to grind a sample until 80 per cent passes a No. 200 sieve, and (*b*) grindabilities in pounds per hour, are plotted against F.W. index numbers. A straight-line correlation in the range of values obtained during the tests exists between F.W. and ball-mill method results.

F.R.L. and Cross Methods:

Figure 3 shows correlations respectively between the F.R.L. and Cross index numbers and the F.W. index.

As pointed out by Selvig[8] and as may be seen from Table I and Figs. 1 and 2, the results of the cooperating laboratories, by the ball-mill and Hardgrove-machine methods agreed within permissible limits. However, the results of the Cross and F.R.L. methods did not. For this reason, only the Cross and F.R.L. averages of the results obtained by the several laboratories are plotted on Fig. 3 to approximate the trend of the correlating curves. It is not unlikely that, had standardized mills such as the U. S. Bureau of Mines ball mill been used for making the tests in accordance with the F.R.L. and Cross procedures instead of the very variable porcelain jar mills, the results obtained by the several laboratories would have agreed more closely. The performance of a jar mill like that of any ball mill depends on its speed, its internal dimensions—especially the internal diameter—the nature of the internal surface, and the dimensions and weight of the ball charge. Differences in any of these characteristics lead to differences in performance. The Cross and F.R.L. methods were probably handicapped by the variability of the several jar mills and by undertreatment of the hard coals due to slippage. The F.R.L. method was probably further handicapped by differences in the pebble charges of the various laboratories.

C.I.T. Roll-Test Method:

Results reported by Yancey and Geer[10] and by Sloman[12] of tests made by them using the C.I.T. roll test on samples of the same five coals tested during the cooperative investigation are included in Table I. There are appreciable differences between the values obtained by Yancey and Sloman for the same coals. In view of this, a correlation between the C.I.T. method and others would for the present be approximate only. The grindability as determined by the C.I.T. method depends to such an important extent on the accurate determination of small amounts of minus 300-mesh material in the product of the test that close checks between laboratories may be difficult to obtain.

Ball-Mill Method with Constant Volume Samples:

Black[11] suggests that the ball-mill method may give more correct ratings of materials if constant-volume, 750-cu. cm. samples are used for the tests instead of constant-weight, 500-g. samples. Yancey and Geer[10] report results of tests on the samples of the five coals tested during the cooperative investigation by modifications of the ball-mill and Hardgrove methods involving the use of constant-volume instead of constant-weight samples. These results are reproduced in Table III. The significance of these results seems to be only that the ratios of grindabilities on the constant-weight basis to corresponding grindabilities on the constant-volume basis are practically the same as the ratios of the weights of the samples. In other words, if a grindability on a constant-volume basis is multiplied by the weight of the sample used in the test and divided by the weight of the sample used in the constant-weight test the result will be practically the same as the grindability determined in the standard constant-weight test after correcting to the standard coal. This is shown in columns *e* and *h* herewith added to Yancey and Geer's[10] original table.

TABLE III.—RELATIVE GRINDABILITIES OF EQUAL VOLUMES AND EQUAL WEIGHTS OF FIVE COALS BASED ON COAL NO. 1 AS 100 PER CENT.

Coal	Weight of 750 cc. of Sample, g.	Ratio $\frac{w_n}{w}$	Relative Grindability					
			Ball-Mill Method			Hardgrove Method		
			Constant Volume	Constant Weight	X[a]	Constant Volume	Constant Weight	Y[a]
n	*a*	*b*	*c*	*d*	*e*	*f*	*g*	*h*
No. 1.....	513	1.02	100	100	100	100	100	100
No 2.....	496	0.99	56	56	56	68	67	67
No. 3.....	516	1.03	49	51	50	58	60	59
No. 4.....	540	1.08	41	44	43	47	51	50
No. 5.....	633	1.27	25	31	31	28	36	35

[a] $X = \frac{b_n c_n}{b_1}$ $Y = \frac{b_n g_n}{b_1}$

where b_n, c_n, g_n = values in the corresponding columns of the table for coals Nos. 1, 2, 3 ...... *n*,

w = weight of the standard sample, and

w_n = weight of the constant volume sample for coal *n*.

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[12] H. J. Sloman, Private Communication, January 15, 1935, to W. A. Selvig, and forwarded to Members of A.S.T.M. Committee D-5, Subcommittee VII.

Inasmuch as capacities of commercial pulverizers are always expressed in weight units and not in volumetric units, the significance of a grindability scale based on constant volume samples is not apparent, even granting that in some mills the volume of coal in the mill at all times, irrespective of the kind of coal pulverized might be more nearly the same than the weight. However, the validity of even this assumption is questionable, because it is known that in some mills the actual coal level maintained in the mill is varied from time to time to suit changes in coals.

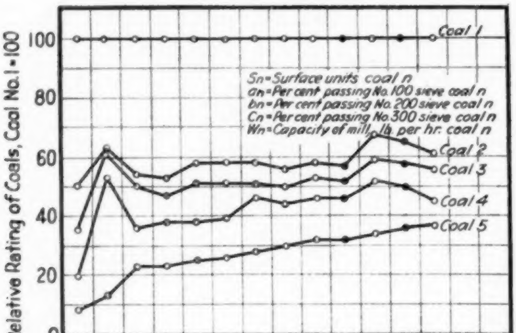

FIG. 4.—Relative Grindability Ratings of the Same Five Coals (Tested During A.S.T.M. Cooperative Grindability Tests) by Various Ranking Methods. (See also Table II.)

APPRAISAL OF GRINDABILITY RATINGS

The grindability of a coal may be arbitrarily defined in a number of ways, as for example: on the basis of calculations of the new surface created (for example, Cross, Hardgrove, C.I.T.); on the basis of the weight or percentage of material produced passing a given sieve (such as, F.W., F.R.L.); on the basis of the amount of work done to grind to a specified fineness (ball mill); or on the basis of the actual capacities obtained in commercial pulverizers.

As shown by Table II and Fig. 4, each of these rating methods unfortunately assigns quite different relative ranks to the same coals. The relative ratings depend not only on the method by which the coal is rated, but also on the coal selected as a standard for reference.

It is then a fair question to ask which of the various rating methods, if any, truly ranks coals as to their behavior in pulverizers.

It is difficult to conclude that any rating method so far proposed truly ranks coals so as to make predictable their effect on the performance of commercial pulverizers. Perhaps, for a pulverizer designed to grind to a certain fineness through a No. 100 sieve, grindability ratings based on the amount of minus 100-mesh material produced during the laboratory test might be more indicative of the probable performance than those based on amounts of surface or minus 200-mesh, or minus 300-mesh product. Likewise, for pulverizers designed to grind to a given fineness through the No. 200 or the No. 300 sieve, grindability ratings based respectively on the amount of minus 200- or 300-mesh material produced in the grindability test might be the best. For it is known[5] that at a given fineness through some one screen, the finenesses through other screens are different for different coals. Also it has been shown [13,14,5] that at a given fineness, the surface per unit weight of coal is not the same for all coals. Consequently, it might well be that for each grinding problem or specification a different grindability scale should be used.

The large variation in the relative rankings of the same five coals by the several methods, scales, or indices as shown on Table II and Fig. 4 is significant. Were a pulverizer to be selected for a given duty on the basis of these rankings without regard to anything else, large errors in the selection could ensue. A pulverizer which would have an indicated capacity of 10 tons on coal No. 1 could be rated at anywhere between 0.8 and 3.7 tons on coal No. 5 depending on which index is used. Even omitting from consideration the Cross and F.R.L. indices, the variation would still be from 2.3 to 3.7 tons. Suppose now it were necessary to select a pulverizer for a capacity of 10 tons on coal No. 5. The size required in terms of its capacity on coal No. 1 could be anywhere between 43.4 and 27 tons. Considering only the two tentative methods[2] the required size would be 31.2 or 27.8 tons, depending on whether the ball-mill or Hardgrove indices are used. As will be shown later, the actual size in terms of its capacity on coal No. 1 will be about 23 tons.

Grindability cannot be used as a quantitative direct index of mill capacity. If the performance of a pulverizer is known on only one coal of known grindability it is not possible to predict its performance on another coal, even though of known grindability. The pulverizer must be first considered and operated as a large-scale grindability testing machine and a sufficient number of coals of widely different grindabilities tested before its performance as affected by grindability can be predicted safely. Only then may its capacity on some particular coal of known grindability not previously tested in the pulverizer be predicted without further test.

WHAT IS GRINDABILITY?

Many engineers believe that "grindability" is an inherent property of a material, and according to Hardgrove[14] if determined on a true surface basis the grindability is independent of the type of pulverizing system used as long as the scavenging is perfect. From this it follows that the capacity of a perfectly scavenged pulverizer grinding different coals to the same specifications under identical conditions will be directly proportional to the grindability as derived by the Hardgrove method. Does this imply that the Hardgrove grindability machine is, or simulates, a perfect pulverizer? During the grindability test the machine is not scavenged at all, nor does the procedure result in the pulverization of all materials to the same specification. Therefore, it is difficult to see why grindabilities derived as prescribed should be proportional to grindabilities on other machines or capacities of pulverizers no matter how well scavenged.

Since, as shown by Fig. 4, no two

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[13] R. A. Sherman, "An Experimental Study of the Burning Characteristics of Pulverized Fuels," *Proceedings*, Third International Coal Conference on Bituminous Coals, Pittsburgh, Pa., November 16, 1931.

[14] R. M. Hardgrove, "The Relation Between Pulverizer Capacity, Power and Grindability," Semi-Annual Meeting, Chicago, Ill., Am. Soc. Mechanical Engrs., Fuels and Steam Power Division, June 25–July 1, 1933.

grindability methods rank coals alike, the question "What Is Grindability?" naturally arises.

It does not appear that any more meaning can be put into the results of a laboratory grindability test than that these results portray the effects of the particular action of the grindability machine on the materials tested under the particular specified test conditions.

Grindability machines are miniature batch pulverizers. No such machine can simulate exactly, except by accident, the action of a pulverizer with continuous feed and discharge at a sensibly uniform rate. It is not even possible to be certain, were a particular standard grindability machine rearranged to operate as a pulverizer with continuous feed and discharge, that the capacity on different coals would be proportional to grindabilities previously obtained on that machine. Obviously, the rate or production of fines to a given specification will be greater with continuous feed and discharge than with intermittent feed and discharge (batch process).

Let us consider in detail the action in the tentative standard grindability machines.

In the Hardgrove test 50 g. of the test sample prepared in the specified way are pulverized in a prescribed manner and length of time. Almost as soon as the test is under way some of the sample will have been pulverized so that it will pass the reference sieve, say the No. 200 sieve. This minus 200-mesh product is not removed and immediately replaced by new feed as in a commercial pulverizer. Therefore, the amount of material available for the production of additional minus 200-mesh material has been reduced by the amount of minus 200-mesh material already produced. Further pulverization of this minus 200-mesh material does not increase the amount of minus 200-mesh material in the mill. The minus 200-mesh product is just made finer and finer. Progressively, as more minus 200-mesh product is produced and not removed and replaced by fresh feed, less plus 200-mesh material is available for the production of additional minus 200-mesh product. Consequently, the net rate of production of new minus 200-mesh product per unit time is constantly decreasing. At first the rate of decrease is not marked because as the test proceeds more and more of the remaining plus 200-mesh sample approaches the minus 200-mesh size and the rate of production of minus 200-mesh product per unit weight of the remaining plus 200-mesh material increases. However, as the test proceeds further, the increase in the rate of production of minus 200-mesh material per unit weight of remaining plus 200-mesh material does not offset the effect of the rapid rate at which the plus 200-mesh material is disappearing. The presence of minus 200-mesh material also tends to cushion the remaining plus 200-mesh material to some extent, and interferes with the free action of the grinding elements on it. This tends to reduce the rate of increase in the production of minus 200-mesh material per unit weight of remaining plus 200-mesh material otherwise occasioned by the progressively increasing average fineness of the plus 200-mesh material. It is certain, therefore, that as a pulverizer with continuous feed and discharge, the Hardgrove grindability machine would produce more 200-mesh material per unit time than as a batch-grinding grindability machine. Furthermore, since each grindability test in the Hardgrove machine is for a fixed time, the harder coals which are pulverized to a lower fineness than the softer coals will be less affected by the action described, and may therefore be ranked somewhat higher by the grindability test than by a true capacity test of the same machine arranged for continuous feed and discharge.

In the ball-mill test a 500-g. sample prepared in the prescribed manner is pulverized in 8 cycles of equal length until 80 per cent of the sample has been removed as minus 200-mesh material; the minus 200-mesh material produced during each cycle being removed before the beginning of the next one. During any one cycle, as more and more minus 200-mesh material is produced, less and less plus 200-mesh material remains in the mill from which to produce additional minus 200-mesh product. At the end of each cycle the minus 200-mesh material produced during the cycle is removed and rejected, and the amount of plus 200-mesh material from which to produce additional minus 200-mesh product during the next cycle is less by the amount of minus 200-mesh material produced and removed. Therefore, as the test progresses, the production of minus 200-mesh material per unit time will be at a decreasing rate, almost imperceptible at first but becoming more and more pronounced. This is shown on Fig. 5, on which are plotted averages of results of the cooperative tests of the ball-mill method. This has been previously explained as a consequence of selective grinding and gradual accumulation of the harder residues as the test progresses. This may be contributory, but actually the rate of production of minus 200-mesh product per unit weight of plus 200-mesh residue is progressively and rapidly increasing because of the increasing average fineness of the plus 200-mesh residue, even though the net rate of production of minus 200-mesh product per unit time is decreasing.

It is true that the removal of the minus 200-mesh material at the end of each cycle, while decreasing the net rate of production of minus 200-mesh material, is of considerable advantage in minimizing cushioning and hence prevents an even more rapid decrease in the net rate of production of minus 200-mesh material as the test proceeds.

If the cushioning effect could be reduced or prevented by removing the minus 200-mesh material as fast as made, the rate of production of minus 200-mesh product would be increased. If the fines thus removed could be replaced by new feed, the rate of production of minus 200-mesh product would still further increase, reaching a constant value after the mill content had become stabilized. An approximation of the probable capacity of the ball mill machine operating thus as a pulverizer with continuous feed and discharge is possible from the data shown on Fig. 5. It is necessary only to make the following assumptions:

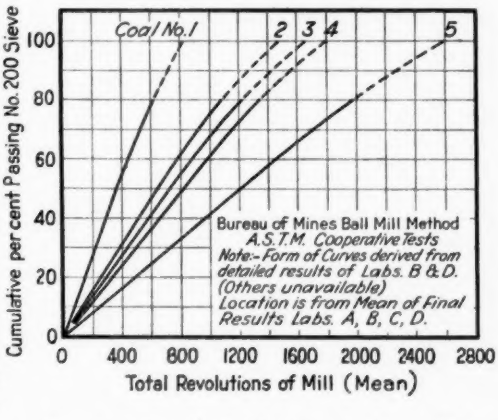

FIG. 5.—Rate of Production of Minus 200-mesh Product per Revolution.

Average results for five coals tested during A.S.T.M. cooperative grindability tests.

1. If at the end of each cycle the amount of minus 200-mesh product removed is replaced by an equal amount of new feed identical to the original charge, the mode of operation of a pulverizer with continuous feed and dis-

charge will be approximated. The approximation will be closer and closer as the length of each cycle is reduced.

2. During each cycle additional minus 200-mesh material will be produced from the fresh feed added at the end of the preceding cycle at the same rate per unit weight of fresh feed as was produced during the very first cycle of the test from the original 500-g. charge.

3. During the second cycle through which the plus 200-mesh first cycle remainder of the added feed passes, the rate of production of minus 200-mesh product per unit weight of remainder will be the same as the rate of production of minus 200-mesh material from the first cycle remainder of the original 500-g. charge during the second cycle of the standard test.

4. During the third cycle through which the second cycle remainder of the added fresh feed passes, the rate of production of new minus 200-mesh material per unit weight of second cycle remainder will be the same as the rate of production of minus 200-mesh material from the second cycle remainder of the original 500-g. charge during the third cycle of the test, and so on.

Granting these assumptions, it is possible to calculate the probable amount of minus 200-mesh material produced, removed, and replaced by fresh feed during each succeeding cycle. If the calculation is extended for a sufficient number of cycles, it is found that the rate of production of minus 200-mesh product becomes constant and the pulverizing process stable. This occurs after the original 500-g. charge has all been replaced and after the size composition of the material in the mill becomes constant.

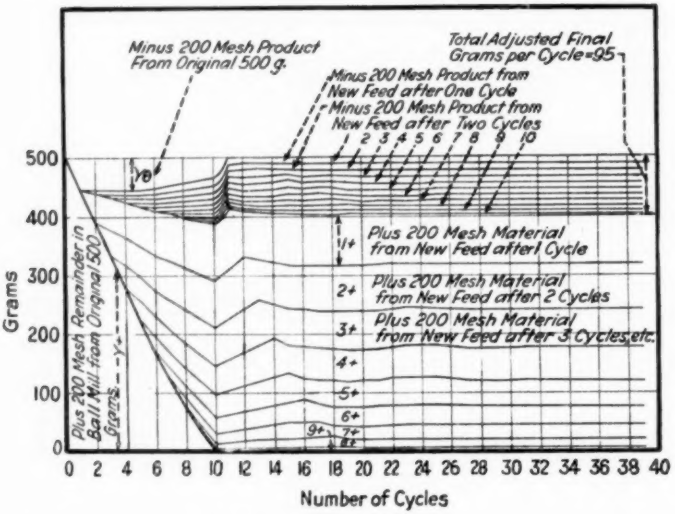

FIG. 6.—Graphical Representation of Calculations for Approximating Capacity of Ball-Mill Grindability Machine If Operated as Continuous Feed and Discharge Pulverizer.

Figure 6 shows graphically the results of such a calculation to determine the probable capacity of the ball-mill machine as a pulverizer on coal No. 4. The same was done for each of the other coals tested. The results are given in Table IV.

This study, which is at best an approximation, indicates that the grindability ball mill should, as a continuous feed and discharge pulverizer, produce between 40 and 50 per cent more minus 200-mesh product than as a batch-grinding grindability machine. The effect of the cycle by cycle removal of fines and replacement by fresh feed tends to increase the spread between the relative rankings of the coals slightly but consistently as the grindability decreases. Were the cycles infinitely short, the continuous constant feed and discharge condition would be almost exactly approximated and the true capacity approached. If the removal of fines as soon as they reach the specified size is continuous, the cushioning effect and the tendency to over-grind the minus 200-mesh material will be absent, the capacity on each coal and the spread between the relative capacities of the different coals will be a maximum for this particular machine. This, however, will not be an absolute index of the relative capacity for any other machine.

A miniature pulverizer like a grindability machine may so under-treat hard materials as compared with the treatment obtainable on the same materials in a particular commercial pulverizer, that the actual commercial pulverizer capacities when compared with the grindability machine results at first appear to increase rapidly with increasing grindability and then, after a critical grindability range is passed, more and more slowly.

Likewise a commercial pulverizer may so under-treat hard materials when compared with the treatment obtainable in the grindability machine that the capacity at first appears to increase slowly with increasing grindability, then more rapidly after a critical grindability range is passed, and again more slowly with further increases in grindability.

It is possible to imagine pulverizers which (*a*) either so absurdly overtreat all materials that the capacity at a given fineness will be practically the same irrespective of the grindability, or (*b*) so absurdly undertreat all materials that none will be pulverized to the required fineness no matter how long treated, that is, will have zero capacity at the specified fineness irrespective of the grindability. Between these extremes can be imagined all sorts of pulverizers with widely varying grindability-capacity relationships.

TABLE IV.—COMPARISON OF PROBABLE CAPACITY OF THE BALL-MILL MACHINE AS A PULVERIZER AND CAPACITIES DETERMINED BY GRINDABILITY TEST.

Coal	Capacity, lb. per hr.		Relative Rating		$\frac{B}{A}$	$\frac{b}{a}$
	As Reported	Adjusted	As Reported	Adjusted		
	A	*B*	*a*	*b*		
No. 1	4.25	6.35	100	100	1.49	1.0
No. 2	2.42	3.55	57	56	1.47	0.98
No. 3	2.20	3.15	52	50	1.43	0.96
No. 4	1.95	2.80	46	44	1.43	0.96
No. 5	1.34	1.90	32	30	1.42	0.94

RELATIVE GRINDABILITY RATINGS FROM COMMERCIAL PULVERIZER TESTS

Commercial Pulverizers as Grindability Machines:

There are commercial pulverizers of some particular design and size located in widely separated plants pulverizing coals of different grindabilities. Sometimes in some one plant a pulverizer is tested with coals of widely different grindabilities. Sometimes, because the source of coal is not constant, a pulverizer must grind coals of varying grindabilities. Unfortunately the capacities obtained from records of such tests or logs of daily operation may not be directly compared because of differences in the moisture, feed size, or fineness of the product during the various runs. Also, the condition of the pulverizer parts may have been better when grinding one coal than another. However, where such data (*a*) include all significant items, and (*b*) were obtained with pulverizer parts in good condition, and where (*c*) a sufficient number of runs on each coal have been made so that statistical averages instead of spot results may be compared, it is possible to reduce all the results to a common standard basis as to moisture, feed size, and fineness with sufficient accuracy to eliminate the effects of these variables in the performance and to distinguish the effect of the particular pulverizing characteristics of each coal on the capacity. The various coals may then be ranked with respect to each other in accordance with the capacities thus obtained, corrected to standard conditions. The ranking thus obtained for a coal may be termed its grindability for the pulverizer in question. If the grindability of the coals pulverized has been determined by some standard laboratory method, a correlation between the laboratory results and the rankings derived from the actual pulverizer capacities may then be established. Thereafter, it would be possible to estimate the performance of the pulverizer on any coal when its standard laboratory grindability is known.

TABLE V.—SUMMARY: PULVERIZER PERFORMANCE WITH COALS OF VARYING GRINDABILITY.

Coal (See Table VII)	Mill Size	Grindability Index			Test Results				Mill Capacity Corrected to Standard Conditions[a]
		F.W. Index	Hardgrove Index, D 409–35 T	Ball-Mill Revolutions, D 408–35 T	Mill Capacity, tons per hr.	Fineness, Per cent Passing No. 200 Sieve	Feed Size, 100 Per cent Passing Ring Diameter, in.	Initial Moisture, per cent	
No. 158......	1070	25.3	104	618	27.50	69.5	¾	2.5	27.2
No. 158......	1070	25.3	104	618	25.50	72.8	¾	2.5	26.9
No. 161......	1070	6.6	41	1750	11.90	81.6	4 mesh	3.2	14.0
No. 158......	1070	25.3	104	618	27.00	69.8	¾	3.0	27.0
No. 160......	1070	6.8	42	1725	12.00	81.0	4 mesh	2.5	13.9
No. 162......	1070	7.0	43	1685	11.10	84.0	4 mesh	2.3	14.3
No. 135......	1120	23.4	101	622	26.50	70.4	¾	4.5	27.0
No. 135......	1120	23.4	101	622	25.30	70.4	¾	4.5	25.8
No. 1006......	750	5.7	37	1930	6.20	84.5	¾	7.0	9.5
No. 115......	750	11.2	61	1185	14.80	71.5	¾	5.1	15.4
No. 113......	772	13.0	67	1055	16.00	71.5	¾	10.9	17.2
No. 113......	750	13.0	67	1055	15.60	71.5	¾	10.9	16.7
No. 176......	620	8.5	49	1450	10.00	75.0	¾	6.4	11.5
No. 128......	650	13.2	69	1040	14.00	72.5	¾	4.0	14.8
No. 128......	620	13.2	69	1040	13.30	70.0	¾	4.0	14.1
No. 185......	490	24.6	103	625	8.20	90.2	¾	5.5	15.7
No. 185......	620	24.6	103	625	10.60	90.2	¾	5.5	19.8
No. 131......	300	14.9	76	935	8.30	81.9	¾	4.4	11.0
No. 150......	300	21.7	97	680	8.80	84.9	¾	3.6	13.1
No. 125......	355	12.0	64	1120	5.70	73.1	¾	10.6	6.8
No. 167......	400	7.8	46	1550	3.00	96.8	3/32	10.6	9.4
No. 167......	300	7.8	46	1550	2.30	96.8	3/32	10.6	7.1
No. 161......	229	6.6	42	1750	2.67	80.0	4 mesh	2.5	3.1
No. 158......	229	25.3	104	618	7.03	70.4	¾	2.5	7.1
No. 180......	229	10.4	57	1250	4.16	73.5	¾	2.8	4.4
No. 140......	242	12.0	64	1120	5.10	81.8	¾	4.5	6.8
No. 140......	180	12.0	64	1120	3.80	81.8	¾	4.5	5.1
No. 122......	180	12.6	67	1075	3.80	80.7	1¼	4.0	5.2
No. 155......	180	17.4	86	815	5.10	75.6	1¼	4.5	6.1

[a] Standard Conditions:
Fineness = 70 per cent Passing No. 200 Sieve.
Feed size = 100 per cent Passing ¾-in. Diameter Ring.
Initial Moisture = 3.0 per cent.

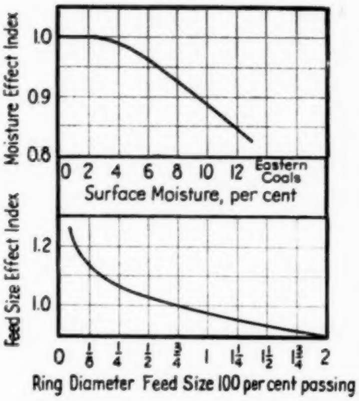

FIG. 7.—Typical Correction Curves for Effect of Moisture and Feed Size on Capacity.

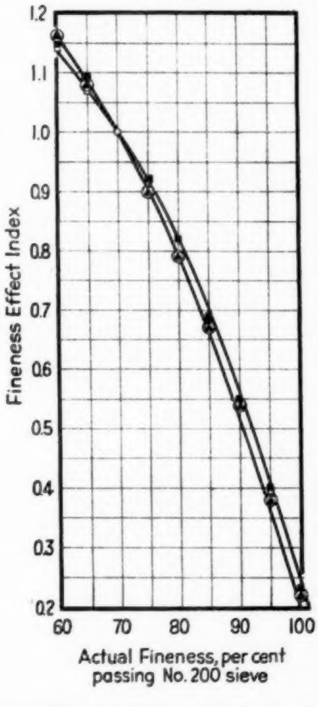

FIG. 8.—Typical Correction Curves for Effect of Fineness on Capacity.

Ball Mills:

Performance data collected since 1931 on ball mills of various sizes, grinding coals of widely varying grindabilities are summarized in Table V. Statistical averages rather than spot points were used, in order to minimize the effects of errors in sampling, weighing, screening etc. These data were all reduced to a common basis, hereafter termed "standard conditions" which are:

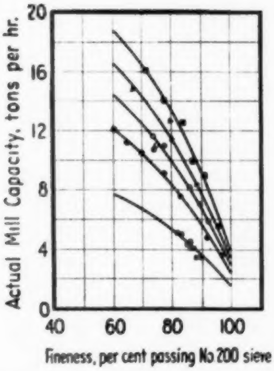

FIG. 9.—Actual Effect of Fineness on Capacity of Commercial Pulverizers of Different Sizes.

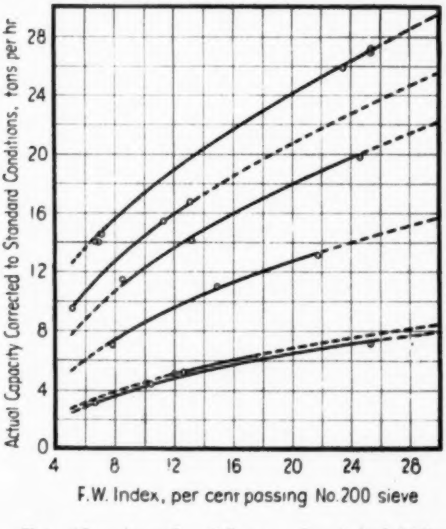

FIG. 10.—Actual Effect of Grindability (F. W. Index) on Capacity of Commercial Ball Mills of Different Sizes, Corrected to Standard Conditions.

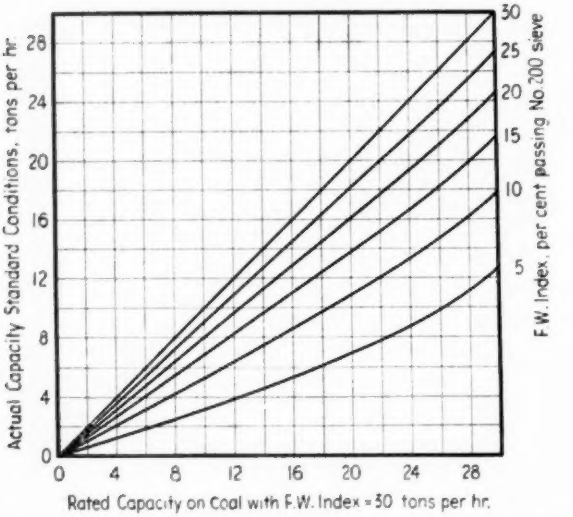

FIG. 11.—Relation Between Actual Capacity and Capacity Rating on Arbitrarily Selected Standard Coal Having F. W. Index = 30 on Coals of Different Grindabilities.

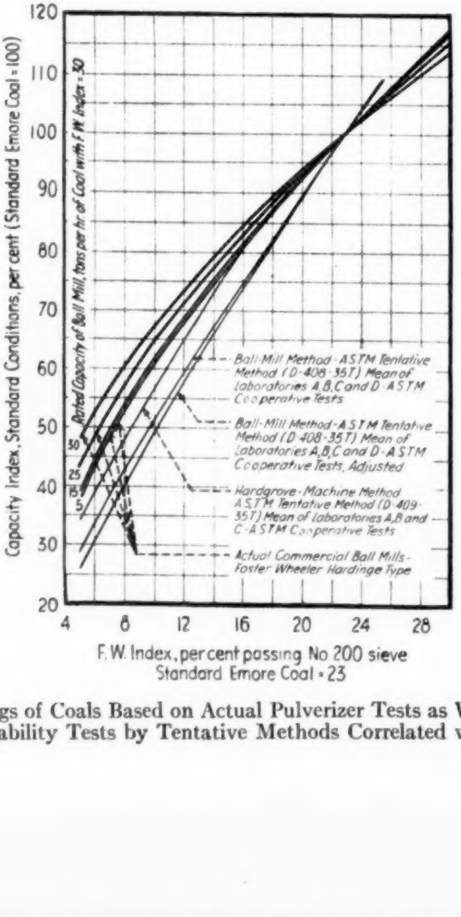

FIG. 12.—Relative Ratings of Coals Based on Actual Pulverizer Tests as Well as Ratings Based on Laboratory Grindability Tests by Tentative Methods Correlated with F. W. Index.

Feed size, 100 per cent passing $\frac{3}{4}$-in. ring,
Surface moisture, 3 per cent, and
Fineness, 70 per cent passing No. 200 sieve.

The great importance of correcting for variations in these factors will be realized after noting the magnitude of their effects on pulverizer capacity.

Figures 7 and 8 show the effects of variations in feed size, moisture, and fineness on the performance of one type of pulverizer. The effect of fineness is so great that accurate correlations are possible only if great care is used in sampling and screening and if a sufficient number of tests under analogous conditions have been made to minimize the effect of spot errors. The derivation of the fineness correction curve is based on statistical studies of the effects of fineness on the performance of many pulverizers. A typical family of fineness-capacity curves for pulverizers of various sizes is shown by Fig. 9. Included in Table V are only statistical averages which represent actual average performances checked over considerable time periods. After reducing the capacities to standard conditions, they were plotted against F.W. index numbers of grindability. As may be seen from Fig. 10, the individual results arrange themselves quite systematically by mill sizes, and by considering the trend of all the points as a whole a family of curves as shown, correlating capacity and laboratory grindability index numbers for each size of pulverizer may be derived. From these, it is possible to determine the relation between rated and actual capacity for a constant grindability. The rated capacity is the capacity under standard conditions on some one coal arbitrarily selected as a standard. Figure 11 shows this correlation. From this the relative capacity on different coals may be calculated with any coal as the standard reference coal, and on Fig. 12 are shown such relative capacity ratings referred to Emore coal, the standard coal of the cooperative investigation. For comparison, relative ratings determined by the tentative methods are also shown on Fig. 12. It is quite significant that the effect of grindability on relative capacity is greatest on the laboratory testing machine, and becomes less and less as the size of the pulverizer increases. Relative ratings of the same coals are higher when based on actual pulverizer tests than when based on laboratory grindability tests. This is probably due (1) to overtreatment in large pulverizers as compared with grindability machines rather than to improper scavenging, because commercial pulverizers employing air separation classifiers are certainly infinitely better scavenged than grindability machines, and (2) to the fact that in the pulverizer there is a

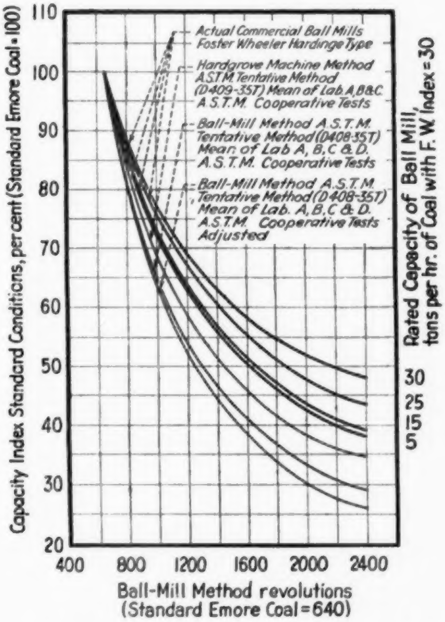

FIG. 13.—Relative Ratings of Coals Based on Actual Pulverizer Tests as Well as Ratings Based on Laboratory Grindability Tests by Tentative Methods Correlated with Ball-Mill Method (D 408 – 35 T) Revolutions.

constant supply of plus 200-mesh material at all times ready to turn into minus 200-mesh product.

On Fig. 13 is shown corresponding correlations between relative pulverizer capacity index numbers and results of grindability tests by the ball-mill method in ball-mill revolutions.

The relation between relative pulverizer capacity index numbers and grindability as determined by the ball-mill method is shown by Fig. 14.

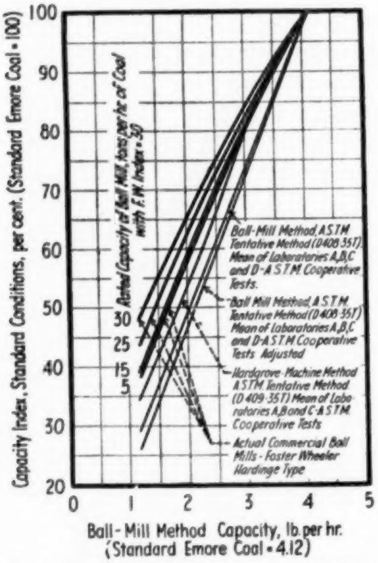

FIG. 14.—Relative Ratings of Coals Based on Actual Pulverizer Tests as Well as Ratings Based on Laboratory Grindability Tests by Tentative Methods Correlated with the Ball-Mill Method (D 408 - 35 T).

The relation between relative pulverizer ratings and grindability as determined by the Hardgrove-machine method is shown in Fig. 15. On this figure a curve based on Fig. 16 summarizing performances of ball-bearing type mills of different capacities from data published by Hardgrove[14] has been added.

The significance of the data shown by Figs. 12, 13, 14, and 15 is that the relative effect of grindability on the capacity of mills of different sizes, though of the same type, is not the same. The smaller the mill, (that is, the grindability machine) the greater the effect. This seems to be clearly true with ball mills of different sizes as compared with each other and with the ball-mill grindability machine.

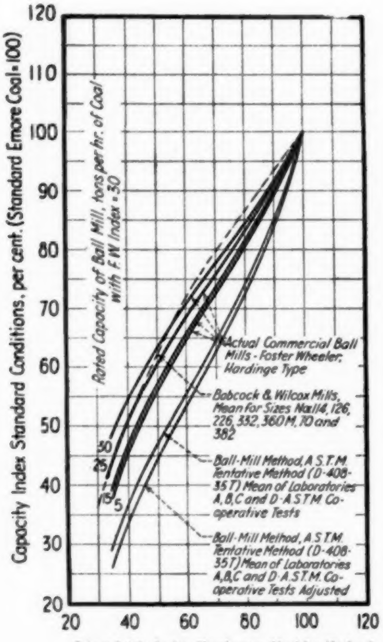

FIG. 15.—Relative Ratings of Coals Based on Actual Pulverizer Tests as Well as Ratings Based on Laboratory Grindability Tests by Tentative Methods Correlated with Hardgrove-Machine Method (D 409 - 35 T) Grindability Index.

Insufficient data on mills of the ball-bearing type were available to the authors to make possible an analysis of the effect of mill size on the relation between capacity and grindability. Possibly because in mills of this type the intensity of treatment may be changed by changing the pressure between grinding balls and races, the effect of mill size may be minimized or eliminated entirely. Nevertheless, as may be seen from Fig. 15, the effect of grindability on ball-bearing type mill capacity is less than relative ratings obtained by the Hardgrove-machine grindability test, although the Hardgrove machine is a miniature ball-bearing type mill.

The relative ratings of the five coals as determined during the cooperative investigation, particularly the two tentative methods, may now be compared with the probable ratings of these coals in actual pulverizers which may be determined from the correlations on Fig. 12, 13, 14, or 15. These are shown in Table VI.

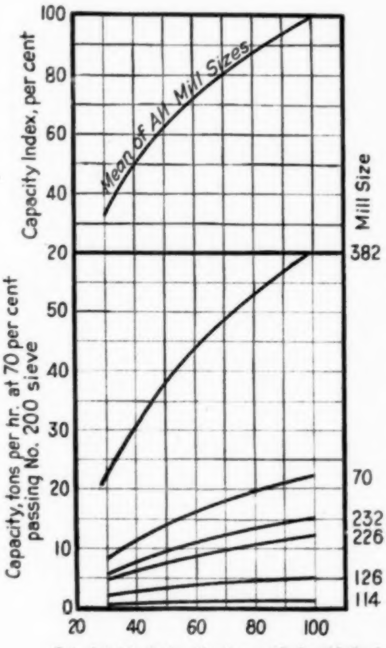

FIG. 16.—Actual Effect of Grindability (Hardgrove Index) on Capacity of Different Sizes of Commercial Mills of the Ball-Bearing Type. See footnote 14.

The significance of the relative ratings in Table VI is that considerable errors can be made in the selection of a mill for some hard coal unless the effect of grindability on the particular size needed is known. For example, relative ratings of coal No. 5 vary between 32 and 49 in terms of coal No. 1, depending on the method used for rating the coal. A mill to pulverize 10 tons of No. 5 coal per hour under "standard conditions" could vary in size as measured by its capacity on coal No. 1 between 31.2 and 20.4 tons per hr. Actually the rated capacity of the mill on No. 1 coal should be approximately 23 tons per hr.

If a rating curve for all mills representing a mean of the values for all sizes is used, it is possible that the smallest mills might be overrated almost 15 per cent on coal No. 5 and largest mills underrated a like amount. On the other hand, if in an effort to play safe all mills are rated on the basis of the smallest mills, the largest mills will be overrated about 25 per cent, which commercially is poor picking.

TABLE VI.—RELATIVE RATINGS OF COALS IN ACTUAL PULVERIZERS COMPARED WITH RATINGS FROM LABORATORY TESTS.

Coal	F.W. Index	Grindability		Ball-Mill Size					Ball-Bearing Type	
		Ball-Mill Method, revolutions	Hardgrove Index	30-ton	25-ton	15-ton	5-ton	Bureau of Mines Grindability Machine	Commercial Pulverizer	Hardgrove Grindability Machine
No. 1	23.8	627	102	100	100	100	100	100	100	100
No. 2	12.7	1077	67	74	73	70	69	57	75	65
No 3	11.2	1219	60	70	68	65	64	52	70	58
No. 4	9.0	1344	51	63	60	57	56	46	62	50
No. 5	5.4	1967	37	49	45	40	39	32	47	36

If the mills were rated in accordance with grindability, the errors would be entirely too large on the hard coals.

CONCLUSIONS

1. The relative ranking of a coal as to its pulverizing characteristics depends

TABLE VII.—PULVERIZING CHARACTERISTICS: AMERICAN AND FOREIGN COALS.

Coal	County	District	Seam or Mine	Proximate Analysis, Dry Basis, per cent					F.W. Index	Hardgrove Grindability Index, D 409 - 35T	Ball-Mill Revolutions, D 408 - 35T
				Volatile Matter	Fixed Carbon	Ash	Sulfur	Calorific Value, B.t.u.			
				Alabama							
No. 101.....	St. Clair	Cahaba	Henry Ellen	34.46	57.01	8.53		14 300	16.8	84	745
				Colorado							
No. 102.....	Huerfano	Walsenburg	Cameron	38.27	43.47	18.26		10 717	9.0	52	1390
No. 103.....	Huerfano	Walsenburg	Laramie	38.13	43.98	17.89		11 662	7.0	43	1685
No. 104.....	Huerfano	Walsenburg	Cameron	36.25	51.38	12.37		12 152	7.8	46	1550
No. 105.....	Huerfano	Walsenburg	Cameron	40.25	51.48	8.27		12 452	10.0	56	1290
No. 106.....	Las Animas	Trinidad	Robinson	32.84	50.55	16.61		12 400	9.0	52	1390
No. 107.....	El Paso	Denver	Stoker's Inlet	52.34	40.33	7.33	0.21	11 217	6.0	39	1865
No. 108.....	El Paso	Denver	Golden Cycle Mill	51.49	39.91	8.60	0.32	11 016	6.0	39	1865
				Indiana							
No. 109.....			Screenings	42.15	47.67	10.18	4.10	12 842	12.8	68	1060
				Illinois							
No. 110.....	Clair		No. 6	41.00	44.00	15.00	4.20	12 150	11.2	61	1185
No. 111.....	Henry	Rock Island	Rock Island	44.20	45.81	9.99		12 720	14.2	74	975
No. 112.....	Vermillion	Danville	Nos. 6 and 7	44.05	44.50	11.45		12 800	11.4	62	1165
No. 113.....	Vermillion	Danville	No. 6	44.00	44.20	11.80		12 600	13.0	68	1055
				Iowa							
No. 114.....	Boone	Des Moines River	Lower Vein	38.09	37.55	24.36	5.76	10 584	14.8	76	940
				Kentucky							
No. 115.....	Pike	Pond Creek	Pond Creek	33.30	60.06	6.64	0.48	14 281	11.2	61	1185
No. 116.....	Harlan	Harlan	Harlan	38.18	55.20	6.62		14 100	9.4	53	1350
No. 117.....	Ohio	West. Ky.		37.50	52.10	10.40		12 000	14.8	76	940
				Ohio							
No. 118.....	Harrison	Pittsburgh No. 8	Stripping	37.61	53.10	9.29		13 400	12.0	64	1120
No. 119.....	Lawrence	Ohio No. 5	Lr. Kittaning	40.40	50.45	9.15		13 620	10.0	56	1290
No. 120.....	Muskingum		Middle Kittaning	40.80	48.35	10.85		12 310	9.6	54	1325
				Pennsylvania							
No. 121.....	Alleghany	Alleghany	Upper Freeport	32.23	58.30	9.47		14 000	16.8	84	845
No. 122.....	Alleghany	Springdale	Upper Freeport	34.00	54.98	11.02	1.13	13 600	12.6	67	1075
No. 123.....	Alleghany		Double Thick Freeport	34.80	54.55	10.65		13 600	14.8	76	940
No. 124.....	Alleghany	Curtisville	Thick Freeport	36.50	58.29	5.21		14 200	11.7	63	1145
No. 125.....	Alleghany	Pittsburgh	Pittsburgh	34.40	60.18	5.42		14 500	14.8	76	940
No. 126.....	Alleghany	Pittsburgh	Pittsburgh	34.77	59.66	5.57	0.96	14 550	12.0	64	1120
No. 127.....	Alleghany	Pittsburgh	Pittsburgh	35.53	53.72	10.75		13 500	13.4	70	1050
No. 128.....	Alleghany	Pittsburgh	Pittsburgh	35.53	53.72	10.75		13 500	13.2	69	1040
No. 129.....	Alleghany	Smithdale	Pittsburgh	34.50	56.60	8.90		13 600	14.8	76	940
No. 130.....	Alleghany		Splint	33.78	58.67	7.55		13 827	7.0	43	1685
No. 131.....	Alleghany	Versailles	Pittsburgh	34.80	58.83	6.37		14 080	14.8	76	940
No. 132.....	Butler	Butler	Upper Freeport	34.72	55.61	9.67		13 500	12.2	65	1100
No. 133.....	Cambria		Lr. Freeport	26.90	66.50	6.60		14 600	26.6	107	605
No. 134.....	Cambria	Cambria	Upper Freeport	26.78	66.70	6.52		14 600	22.7	99	655
No. 135.....	Cambria	Cambria	Upper Freeport	26.78	66.70	6.52		14 600	23.4	101	622
No. 136.....	Cambria		Lr. Kittaning	19.09	53.54	27.37	5.94	10 859	24.2	102	630
No. 137.....	Clarion	Lr. Kittaning	Lr. Kittaning	35.50	53.65	10.85		14 000	12.2	65	1100
No. 138.....	Clarion	Lr. Kittaning	Lr. Kittaning	33.59	52.08	14.33	4.17	12 759	12.6	67	1075
No. 139.....	Clarion	Lr. Kittaning	Lr. Kittaning	36.21	50.27	13.52	4.66	12 916	12.6	67	1075
No. 140.....	Clarion	Lr. Kittaning	Lr. Kittaning	37.14	51.75	11.11	3.50	13 395	12.0	64	1118
No. 141.....	Clearfield	Reynoldsville	Lr. Kittaning	39.29	51.85	8.86	2.74	13 920	11.7	63	1145
No. 142.....	Clearfield	Moshannon	Lr. Freeport	30.20	62.30	7.50	1.45	14 450	20.3	94	715
No. 143.....	Elk	Shawmut	"B"	31.62	58.24	10.14		13 820	18.0	88	795
No. 144.....	Fayette		Pittsburgh	34.20	58.80	7.00	0.9	14 250	12.7	67	1075
No. 145.....	Indiana	Lr. Kittaning	Waterman No. 2	22.56	49.84	27.60		10 619	25.4	105	615
No. 146.....	Indiana	Lr. Kittaning	E - R Mine	25.51	46.73	27.76		10 567	18.0	88	795
No. 147.....	Indiana	Lr. Freeport	Lr. Freeport	30.13	58.82	11.05	2.78	13 861	21.2	96	690
No. 148.....	Indiana		Lr. Freeport	27.62	55.06	16.69	3.51	12 569	22.0	98	670
No. 149.....	Indiana	Indiana	Upper Freeport	28.57	64.20	7.23		14 561	22.0	98	670
No. 150.....	Indiana	Indiana	Upper Freeport	28.66	63.70	7.64		14 489	21.7	97	680
No. 151.....	Indiana	Indiana	Moween	29.00	58.60	12.40		13 550	19.2	91	745
No. 152.....	Indiana	Lr. Kittaning	Lr. Kittaning	25.98	66.95	7.07		15 000	22.7	99	655
No. 153.....	Jefferson	Adrian	Lr. Freeport	30.40	63.46	6.13	1.91	14 500	22.0	98	670

TABLE VII.—*Concluded.*

Coal	County	District	Seam or Mine	Proximate Analysis, Dry Basis, per cent					F.W. Index	Hardgrove Grindability, Index, D 409 - 35T	Ball-Mill Revolutions, D 408 - 35T
				Volatile Matter	Fixed Carbon	Ash	Sulfur	Calorific Value, B.t.u.			
			PENNSYLVANIA—*Concluded*								
No. 154.....	Jefferson	Adrian	Lr. Freeport	28.18	67.60	4.22		15 000	22.0	98	670
No. 155.....	Jefferson	Adrian	Lr. Freeport	31.29	59.53	9.18	1.53	14 013	17.4	86	815
No. 156.....	Jefferson		Lr. Freeport	30.08	60.22	9.70	0.92	13 894	19.8	93	735
No. 157.....	Jefferson	Kramer	Lr Freeport	28.60	65.78	5.62		14 350	21.2	96	690
No. 158.....	Somerset		Miller	18.90	72.40	8.70			25.3	104	618
No. 159.....	Westmoreland	Pittsburgh	Pittsburgh	34.50	55.70	9.80		13 610	16.3	82	865
No. 160.....	Lancaster	Holtwood	River Anth.	71.8	72.75	20.07	0.06	11 260	6.8	42	1725
No. 161.....	Lancaster	Holtwood	River Anth.	71.8	72.75	20.07	0.06	11 260	6.6	41	1750
No. 162.....	Lancaster	Holtwood	River Anth.	71.8	72.75	20.07	0.06	11 260	7.0	43	1685
No. 163.....			Anth. Fines	4.16	72.94	22.90	0.90	11 300	6.0	39	1865
No. 164.....			Anth. Fines	4.16	73.07	22.77	0.90	11 275	5.9	38	1885
No. 165.....			Anth. Fines	6.70	82.80	10.50	0.80	13 175	9.6	54	1325
No. 166.....			Anth. Fines	5.14	82.46	12.40	0.60	12 805	5.9	38	1885
No. 167.....			Anth. Fines	5.92	82.63	11.45	0.70	12 988	7.8	46	1550
No. 168.....	Northumberland	Anth.	Bear Valley	6.6	83.8	9.6	0.9	13 860	9.0	52	1390
No. 169.....	Schuylkill	Anth.	St. Nicholas	3.0	87.6	9.4	0.5	13 470	5.4	36	1990
			UTAH								
No. 170.....	Carbon	Castlegate	Castlegate	45.74	47.64	6.62	0.63	13 486	9.6	54	1325
			WEST VIRGINIA								
No. 171.....	Brooke	Panhandle	Pittsburgh	39.12	50.74	10.14	3.78		12.0	64	1120
No. 172.....	Brooke	Panhandle	Pittsburgh	38.89	50.75	10.36	3.14	13 310	11.4	62	1165
No. 173.....	Fayette	New River	Sewell	24.79	60.03	15.18		13 100	18.0	88	795
No. 174.....	Kanawha	Kanawha	Powellton	32.01	63.50	4.49	1.08	14 700	15.8	80	895
No. 175.....	Kanawha	Kanawha	Coalburg	33.20	55.45	11.35	0.80	13 050	10.0	56	1290
No. 176.....	Kanawha	Kanawha	Coalburg	32.05	51.99	15.96	0.70	12 248	8.5	49	1450
No. 177.....	Kanawha	Kanawha	Winifrede	31.55	61.35	7.10	0.86	14 150	13.8	72	1000
No. 178.....	Kanawha	Kanawha	Powellton	36.85	58.30	4.85	0.64	14 250	11.0	60	1200
No. 179.....	Marion	Rivesville	Sewickley	36.73	54.87	8.40	2.21	13 800	12.3	66	1095
No. 180.....	Marion		Pittsburgh	36.90	54.30	8.90			10.4	57	1250
No. 181.....	McDowell	Pocahontas	No. 3	18.50	76.00	5.50	0.5	14 880	23.8	102	618
No. 182.....	Monangalia	Maiden	Pittsburgh	35.35	55.35	9.30		14 600	15.8	80	895
No. 183.....	Monangalia	Pursglove	Sewickley	35.33	55.95	7.18		13 870	15.8	80	895
No. 184.....	Monangalia	Fairmont	Waynesburg	35.38	50.30	14.32		12 500	15.8	80	895
No. 185.....	Raleigh		Beckley	19.60	75.50	4.90			24.6	103	625
No. 186.....	Webster		Sewell	30.40	62.82	6.78		14 650	30.0	112	590
No. 187.....	Westmoreland	Valley Camp	Thick Freeport	35.79	54.41	9.80		13 780	15.8	80	895
			WYOMING								
No. 188.....	Sweetwater	Union Pacific	Rock Spring	43.24	51.26	5.50	0.97	12 748	10.3	57	1265
			CHILE								
No. 189.....		Lirquen	Run of mine	38.00	48.97	13.03		11 651	3.6	28	2555
No. 190.....		Lota	Run of mine	37.57	54.05	8.38		13 131	4.6	33	2200
No. 191.....		Schwager	Run of mine	37.63	56.49	5.88		13 595	4.6	33	2200
			BRITISH COLOMBIA								
No. 192.....	Vancouver		Sunrise	45.90	42.10	12.00	0.71	11 600	7.0	43	1685
No. 193.....	Vancouver		Sunrise	39.70	49.99	10.31		11 560	7.2	44	1640
No. 194.....	Vancouver		Pleasant Valley	40.65	54.33	5.02	0.58	12 480	6.0	39	1865
			WALES								
No. 195.....		Anth.	Screenings	7.75	88.25	4.00	0.94	14 655	8.5	49	1450
No. 196.....		Anth.	Screenings	7.77	87.97	4.26	0.97	14 661	9.0	52	1390
			COKE								
No. 1000....	Crude Still Coke—6 per cent oil			14.84	82.52	2.64	1.80	15 914	18.5	89	775
No. 1001....	Cracking Oil Coke—1.52 per cent oil			12.72	86.29	0.99	1.14	15 171	16.8	84	845
No. 1002....	Petroleum Coke			3.74	95.31	0.95		14 689	8.5	49	1450
No. 1003....	Petroleum Coke			50.15	49.45	0.40	10.58	13 200	15.8	80	895
No. 1004....	Coke Breeze			1.41	88.12	10.47		12 707	5.0	34	2075
No. 1005....	Coke Breeze			1.41	88.12	10.47		12 707	5.7	37	1930
No. 1006....	Coke Breeze			0.91	90.51	8.58		13 090	5.3	36	2010
No. 1007....	Coke Breeze			2.54	87.49	9.97		12 345	5.3	36	2010
No. 1008....	Coke Breeze			1.16	87.11	11.73		12 520	7.0	43	1685
No. 1009....	Coke Breeze			1.43	86.21	12.36		12 647	6.0	39	1865
No. 1010....	Coke Breeze			0.91	90.59	8.50	0.51	13 100	5.3	36	2010
No. 1011....	Coke Breeze			0.87	86.83	12.30	0.60	12 660	5.3	36	2010

on the method used. No two methods assign the same relative rank to the same coal.

2. The two tentative methods assign quite different relative ratings to the same coal. But these ratings may be correlated in view of the permissible accuracy with which tests may be checked and reproduced by these methods.

3. Relative ratings derived from properly standardized tests of commercial pulverizers rank coals closer together than the standard laboratory tests.

4. Pulverizers of different sizes, though of the same type, do not rank coals alike.

5. Laboratory grindability ratings are useful, and may be used to predict the performance of a pulverizer on a coal of known grindability without test, but only provided the grindability capacity relationship for the pulverizer has been previously determined by test, and effects of such other factors as feed size, moisture, and fineness, tending to mask the effect of grindability, have been properly taken into consideration.

6. A large number of coals tested by the F.W. method can now be correlated with grindabilities obtained by other methods. A partial table of coals tested by the F. W. method giving the corresponding grindabilites by the Tentative Methods is given in Table VII.

Acknowledgments:

The authors are indebted to H. F. Yancey, Supervising Engineer, Northwest Experiment Station, U. S. Bureau of Mines, Seattle, Wash.; to R. E. Gilmore and C. E. Baltzer, Canada Department of Mines and Resources, Fuel Research Laboratory; and R. M. Hardgrove, Babcock & Wilcox Co., New York City, for furnishing additional laboratory data for the coals tested during the A.S.T.M. cooperative investigation and for their helpful suggestions; and to V. J. Riggio and A. Koch of the Foster Wheeler Corp. for their assistance in the preparation of the data, curves, and manuscript.

DISCUSSION

Mr. R. M. Hardgrove[1] (*presented in written form*).—The authors have presented a paper which is of considerable value in understanding the relation between grindability and pulverizer capacities.

I do not believe that any useful purpose has been served by setting up a new scale of grindability which is different from either one of the two now in use as tentative standards. The author has pointed out that pulverizer characteristics must be known and their relation to any one of the grindability scales established from experience before they can be applied usefully. Therefore, if these scales are arbitrary units, there seems to be no advantage in setting up a third scale that is even more arbitrary than the present two.

We wish heartily to endorse the discussion on volumes *versus* weights and the illustration pointed out in Table III. We still buy our coal by the ton and pulverizer performance is still based on tons pulverized and not on a cubic foot or cubic yard basis.

The authors use the Hardgrove machine, but have set up a procedure in which 75 revolutions are used instead of the standard 60. They then proposed that there is cushioning taking place in the grindability test of a batch nature. In our early experiments, we determined when cushioning began to be felt in the results and stopped our grinding operation short of where this would be felt on the softest coals tested. We, therefore, had a valid reason for choosing 60 revolutions, and the real difference between the authors' procedure and the standard lies in the fact that the larger number of revolutions does result in some cushioning on the very highest grindability coals.

The effect of grindability being different with various sizes of tube mills is very interesting and we suspect that it may be caused by a difference in the efficiency of these sizes of mills. In the writer's paper in 1933,[2] a large amount of data was presented on a wide variety of sizes of ball-bearing pulverizers. This showed that the size of the pulverizer had no effect on the relation between grindability and capacity, but it is also true that these pulverizers have a practically constant efficiency and power consumption irrespective of size.

Figure 15 is, of course, the meat of the paper and it is gratifying to note that the average correction for grindability is not much different for the tube mills and the B. & W. pulverizers, especially if the curves are made to coincide at 55 grindability corresponding to the average for Pittsburgh No. 8 coal.

The practical application of the established grindability information on various types and sizes of pulverizers is of the utmost importance to all interested parties and we appreciate the amount of information which has been added to the published data by means of this paper.

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[1] Engineering Design, The Babcock & Wilcox Co., New York City.

[2] R. M. Hardgrove, "The Relation Between Pulverizer Capacity, Power and Grindability," Semi-Annual Meeting, Chicago, Ill., Am. Soc. Mechanical Engrs., Fuels and Steam Power Division, June 25–July 1, 1933.

MR. J. F. BARKLEY[3] (*presented in written form*)[4].—The authors emphasize the needs of the designer who is is responsible for furnishing grinding equipment that will give the most economical results, and who must work to the greatest possible accuracy to meet commercial requirements. Considering the complex nature of coal, it is not surprising that laboratory tests on grindability cannot be translated or interpreted directly with sufficient accuracy to meet the designer's needs. With the many types of pulverizers available and considering the varied nature of coal, it appears quite logical that it would be necessary to make large scale tests before laboratory tests can be correlated. This paper gives much interesting information in this regard.

There is another type of worker in fuel utilization, however, who does not necessarily require great accuracy, but who could use some pertinent information. The ordinary user of pulverizers, particularly the smaller user, always must keep in mind the possibility of saving money or improving operation by changing to a different coal. If he has some laboratory expression of the "grindability" of a coal, he can frequently decide immediately whether or not he wants to try out such a coal. A laboratory grindability test gives him some idea of whether the coal is harder or easier to grind than the one he is now using. He is not faced with the necessity of making a final decision from such a laboratory expression as is the designer when he decides on the size of his pulverizer. The coal user would take perhaps several coals whose grindability is within the range of what he might think desirable and try them out. Proper use of a laboratory grindability index should result in his testing a much smaller number of coals with the attending expense and difficulty, his final decision being made on the plant test itself.

MR. R. A. SHERMAN.[5]—The conclusions of the authors are somewhat disappointing in that, after the development work that has gone into the tests for grindability, it is necessary to conduct grindability tests on the various types of mills before one can use the grindability ratings obtained by the laboratory tests. They infer that this is inherently true of any grindability tests, and they do not appear to hold out any other hope for any other method. Do the authors believe that there is any need for further work on the subject of grindability methods in the hope that one could obtain an index that could be used directly without the necessity of confirming mill tests?

MESSRS. MARTIN FRISCH[6] AND A. C. FOSTER[6] (*authors' closure, by letter*).—Mr. Hardgrove's discussion is of special interest because of his long interest in the subject of grindability measurements and his valuable contribution to the art.

The authors agree with Mr. Hardgrove that a new scale of grindability is not needed, but they regret that he so misread the paper as to conclude that a new scale is being proposed. The F.W. scale is utilized in the paper only as a parameter for correlating the two A.S.T.M. tentative standard scales with actual pulverizer performances. This could be done, *first*, because the pulverizer performances available to the authors and presented in the paper had previously been correlated with the F.W. grindability scale, and *second*, because after the A.S.T.M. cooperative investigation the F.W. scale and the A.S.T.M. standard scales were accurately correlated after parallel tests on identical coals.

The real purpose of the paper was *first*, to show that grindability no matter

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[5] Fuel Engineer, Battelle Memorial Institute, Columbus, Ohio.

[6] Chief Engineer, and Pulverizer Engineer, respectively, Boiler and Pulverizer Division, Foster Wheeler Corp., New York City.

by what method derived or how expressed ranks coals arbitrarily as to their relative pulverizing characteristics, and *second*, to show how actual commercial pulverizer performances may be correlated with the two arbitrary ranking scales defined by the present A.S.T.M. tentative grindability standards.

With reference to Mr. Hardgrove's comment to the effect that because in the F.W. method utilizing a grindability machine of the Hardgrove type running for 75 revolutions instead of 60 as called for by the standard test, cushioning is more likely to occur than in the standard tests, reference is invited to Table I. A study of the table will show that the F.W. method spreads the rankings of the coals tested farther apart than the standard Hardgrove test. This indicates less cushioning if anything. As a matter of fact, when the F.W. machine was calibrated it was tested at 60 revolutions as well as 75. At 60 revolutions the F.W. machine did not give as consistent results as at 75. Quite possibly 60 revolutions is best for the standard machine.

Mr. Hardgrove expresses the opinion that the capacity-grindability relationship is not the same for different sizes of tube mills as shown by Fig. 15 because of differences in the efficiencies of tube mills of different sizes, but that for mills of the ball-bearing type the grindability-capacity relationship is the same irrespective of size because all sizes are equally efficient. A study of Figs. 15 and 16, and Mr. Hardgrove's own paper from which he quotes and from which came the data for Fig. 16 indicates:

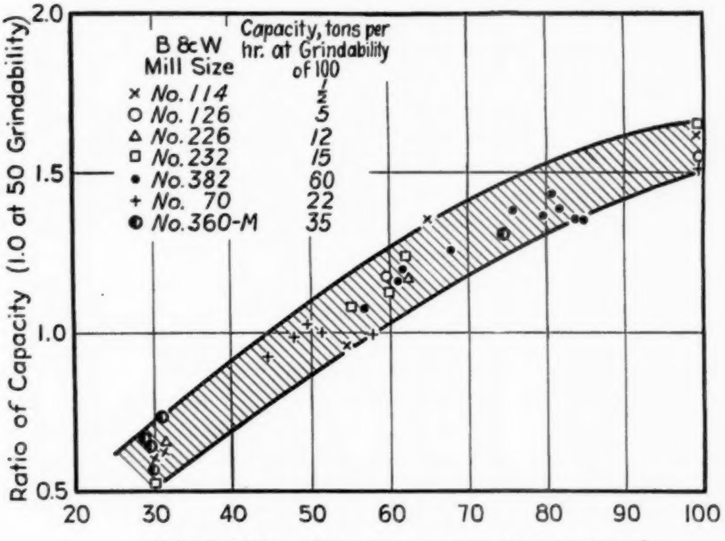

FIG. 1.—Actual Effect of Grindability on Capacity of Different Sizes of Commercial Mills of the Ball-Bearing Type.

(From Fig. 16 of paper by R. M. Hardgrove.[2])

1. With ball mills of the type referred to in the paper and specifically in Fig. 15 the larger the mill the less the effect of grindability. The capacity index ratios at various grindabilities for a 30-ton and a 5-ton mill are as follows:

GRINDABILITY	CAPACITY INDEX 30-TON MILL / CAPACITY INDEX 5-TON MILL
40	1.17
60	1.09
80	1.07
100	1.0

2. On the basis of his own data Mr. Hardgrove is not justified in concluding that for ball-bearing mills the grindability-capacity relation is not affected by size of mill. The accompanying Fig. 1 shows the actual test points from Mr. Hardgrove's paper used for deriving the topmost curve of Fig. 16 in the paper which shows the mean relation between grindability and capacity index of many sizes of ball-bearing mills. The points fall in a band of considerable width and indicate that either the results of the tests of the mills of various sizes were not sufficiently accurate to distinguish the size effect or to justify a single capacity grindability curve applicable to mills of all sizes. Actually the spread between maximum and minimum capacity index at any grindability is greater for the ball-bearing mill data cited by Mr. Hardgrove than is the spread shown in Fig. 15 and in (1) above because of variations in ball-mill sizes.

GRINDABILITY	MAXIMUM CAPACITY INDEX / MINIMUM CAPACITY INDEX
40	1.34
60	1.22
80	1.17
100	1.10

Mr. Barkley makes the excellent point that the user of coal as distinguished from the pulverizer designer needs a satisfactory means for ranking coals as to their pulverizing characteristics to guide him in his coal purchases. Either one of the adopted A.S.T.M. standard test methods can be profitably used by coal purchasers to guide them in their coal selections. However, the purchaser must avoid the conclusion that a coal with a grindability 50 per cent greater than that of some other coal will result in a 50 per cent increase in pulverizer capacity. He must test his particular equipment carefully to find out just what the increase will be. If he does this for several coals he may derive a correlation which will enable him to appraise future coal offerings as to their pulverizing characteristics without further test.

Mr. Sherman raises the question as to whether a method for rating coals is in sight which could be used directly for predicting the pulverizing characteristics without confirming mill tests. The authors do not believe the development of such a test is probable. However, if such a test were possible it would eliminate a lot of guess work from pulverizer design and operation.

Mr. Sherman also asks whether further work on the subject of grindability methods is advisable. The authors believe that much may yet be done to simplify grindability testing. It is our opinion that nothing but confusion follows the expression of grindability on a percentage basis. So many people take the easy interpretation, which at one time was encouraged, that mill capacity and grindability are proportional. It would, in our opinion, be better to use the results of the laboratory test as the grindability index instead of some calculated number as at present. The results of the tentative standard test method could well be expressed as follows:

1. Grindability by Hardgrove machine method is equal to the percentage through the 100, 150, 200 or 300-mesh screen. The screen to use is to be adopted and specified by the A.S.T.M. after further study.

2. Grindability by the ball-mill method is equal to the number of revolutions required to reduce the specified sample so that 50, 60, 70 or 80 per cent will pass through a 200-mesh screen. The fineness to use is to be determined and specified by the A.S.T.M. after further study.

RÉSUMÉ OF SYMPOSIUM ON CORRELATION BETWEEN ACCELERATED LABORATORY TESTS AND SERVICE TESTS ON PROTECTIVE AND DECORATIVE COATINGS

ABSTRACTED BY A. C. ELM[1]

A compilation of the papers and discussion presented at the symposium have been issued as a special publication entitled Symposium on Correlation Between Accelerated Laboratory Tests and Service Tests on Protective and Decorative Coatings.—ED.

A feature of the 1937 Annual Meeting of the American Society for Testing Materials was a symposium on the correlation between accelerated laboratory tests and service tests on protective and decorative coatings. This symposium, held June 29, was conducted by Subcommittee VII on Accelerated Tests for Protective Coatings of the Society's Committee D-1 on Paint, Varnish, Lacquer and Related Products.

In opening this symposium, the chairman of the committee in charge of the program, Mr. H. A. Nelson, Assistant to General Manager, Technical Dept., The New Jersey Zinc Co., Palmerton, Pa., called attention to the fact that it is about 25 yr. since Muckenfuss described the first attempt to study the application of an accelerated testing scheme to paint products intended for exterior service. Since then a wide variety of accelerated laboratory tests have been developed which are now in daily use and it seems that some effort to take stock of their proper position in the industry is in order. Realizing that the problem is of even more vital importance today because improvements in raw materials are opening up new possibilities for better paint products at a bewildering rate, a more rapid evaluation of protective and decorative coatings must be approached from many different angles. These depend on the nature of the protective or decorative coatings and the uses for which they are intended, so the Symposium was not limited to a discussion of so-called accelerated weathering tests but comprised three topics: namely, (I) Finishes for Indoor Service, (II) Paints for Exterior Service on Wood, and (III) Paints for Iron and Steel.

Topic I—Finishes for Indoor Service:

Mr. Wayne R. Fuller, Manager of Industrial Research, Devoe & Raynolds Co., Inc., Louisville, Ky., introduced Topic I and briefly reviewed the development of the large number of accelerated testing methods employed in the paint industry. Since, in the main, these methods are the outcome of the efforts of various laboratories working individually, most tests lack standardization of method, technique and apparatus. Certain methods have been used for years without any thorough-going attempt to determine the significance of the test results. The only sound viewpoint is that the method should be

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[1] Investigator, Paint Section, Research Division, The New Jersey Zinc Co., Palmerton, Pa.

carefully tested and standardized before it is adopted as a regular procedure.

Accelerated tests may be broadly classified under two types:

1. Those which undertake to reproduce in a single test all of the factors encountered under service conditions, and

2. Those which aim to study each property separately.

The former group includes accelerated weathering tests; the latter distensibility tests of films which have been subjected to accelerated aging. Another form of accelerated testing involves aging the film under normal conditions and applying testing methods which reveal film changes before they would be apparent otherwise.

Regardless of the type of test, careful attention should be given to such points as control of film thickness and conditions during aging and testing of the films. Tests should be conducted at regular intervals of aging and extending over a sufficient period of time to indicate the changes that occur with age, and hence, ultimate performance. In the development of testing equipment, care should be taken that the refinement will not be carried to the point that the cost will be prohibitive to all but a few laboratories.

Mr. C. C. Hipkins, Member of Technical Staff, Bell Telephone Laboratories, Inc., New York City, discussed the relationship between accelerated tests and service behavior of finishes for telephone apparatus. Although the environmental factors leading to film failure on finishes used indoors are very mild as compared to outdoors, mechanical deforming stresses such as impact or flexing may shorten the life of these finishes greatly. Based on the assumption that the quality of an organic finish to be used indoors is definable in terms of measurable and significant physical attributes, it has been possible to obtain a valid definition of durability. The effective strength of a finish is determined by a combination of abrasion and distensibility tests and its adherence and resistance to sudden mechanical shock by suitable impact tests conducted under controlled conditions of film thickness, temperature and humidity. By repeating the physical tests periodically after intervals of exposure to either drastic or mild environments, definitely reproducible changes in character with time are observed. The results of several years' work have shown that high rating on these physical tests coupled with retention of high values, serves as a satisfactory measure of durability.

When an interior finish must withstand mechanical abuse, the physical tests offer a direct means for selection. In numerous cases where equipment is subjected to repeated and severe mechanical forces, such as repeated drops, impact tests gave a practically perfect correlation with simulated service tests and actual field behavior. At times, certain finishes must withstand additional specific requirements such as the effect of perspiration, battery room acids or other chemical influences. In such cases special accelerated tests for these properties are utilized, but it must be kept in mind that, as a rule, very little may be sacrificed in the inherent physical properties.

Mr. Leo Roon, Technical Director, Roxalin Flexible Lacquer Co., Inc., Elizabeth, N. J., emphasized the necessity of basing accelerated tests of any kind on an exact knowledge of the conditions to which the finish is to be exposed during actual service. In an effort to differentiate between suitable and unsuitable finishes, consumers of organic coatings frequently set up accelerated tests which apparently bear some relation to the actual service required, whereas actually they have no significance. In one case, for example, finishes which were formulated to withstand the prescribed test for alcohol resistance were found to give very inferior service because no consideration had been given to abrasion resistance and other similar properties. In a second example, a so-called accelerated perspiration test consisting of exposure to oleic acid and various salt solutions yielded results impossible of interpretation in terms of actual service. These and other similar case histories led to the conclusion that accelerated tests are of little value unless they duplicate closely the actual service conditions and are founded on a thorough understanding of every phase of the services to which the finished articles are to be subjected.

Mr. J. B. Bullitt, Jr., Chemist, Finishes Division, Philadelphia Laboratory, E. I. du Pont de Nemours and Co., Philadelphia, Pa., described his experiences with a number of accelerated tests for the determination of the probable service behavior of white interior finishes. During the past few years considerable data have been accumulated on the accelerated testing of white interior metal finishes suitable for use on refrigerators and other similar equipment. Consideration is given to only four of the more important characteristics of interior finishes: namely, (1) hardness and flexibility as a measure of resistance to mechanical abuse, (2) rust and humidity resistance, (3) color retention, and (4) grease resistance.

Although the usual impact, bend and scratch tests, particularly when accompanied by accelerated aging at slightly elevated temperatures, correlate quite well with the tendency of the finish to chip in ordinary indoor service, scratch and indentation hardness have not correlated well with normal marring and scarring in service. Laboratory exposures in a high humidity cabinet give a satisfactory indication of blister resistance, but correlation with rusting is less satisfactory, since the humidity tests do not give the same type of rusting that is encountered in service. The salt-spray test, although widely used by the trade, is of slight value, since it is difficult to obtain duplicate results and to interpret them in terms of actual service. It seems best to test this property by exterior exposure, preferably in Florida. Color retention tests are of some value when interpreted with great care and when testing finishes of the same type, but are of no value in comparing different classes of finishes. It is best to base conclusions on exposure under conditions simulating actual service. Grease resistance and perspiration resistance are perhaps the most difficult properties to measure in an accelerated test. In general, the less accelerated the test the more reliable it is, but even when making no attempt to accelerate the test, it has been impossible to reproduce exactly in the laboratory some types of failure obtained in service. Nevertheless, a combination of a variety of tests with different greases, different types of exposures, etc., will give a fairly reliable picture of the expected performance.

Mr. W. H. Lutz, Technical Director, Pratt & Lambert, Inc., Buffalo, N. Y., discussed accelerated testing of furniture lacquers. In order to reproduce the normal deterioration of furniture finishes, the weathering factors consisting of moisture and temperature changes and sunlight were combined into an accelerated weathering cycle, comprising 6.2 per cent of light at low humidity, 4.2 per cent of light at high humidity, 10.4 per cent of refrigeration and 79.2 per cent of normal room exposure. It will be noted that only a little more than one-fifth of the cycle consisted of accelerating influences. The normal periods between periods of acceleration were found necessary to reduce the indirect type of failure. Although the experience with this test cycle is limited and experimental data are insufficient to support extensive claims for its reliability, it may be stated that fair correlation between this accelerated exposure and normal service conditions can be obtained.

Mr. Wayne C. Norris, Resin Development Laboratory, American Cyanamid and Chemical Corp., New York City, discussed the resistance to cold checking of lacquers and other surface coatings and also described an apparatus and method for evaluating this property. One of the important factors involved in the evaluation of the serviceability of almost all protective and decorative finishes is the ability to withstand sudden temperature changes. Since this has been fairly well recognized it has become an accepted part of exposure cycles.

After due consideration of the factors involved in a test of this nature, equipment was designed which can be used to give a very high rate of cooling to panel surfaces, and is thermostatically controlled to maintain temperatures from −7 to −34 C. within limits of ±2 deg. Cent. A complete description of this equipment is given by the author in a paper presented at the meeting.[2] The end point of the test consists of transverse or cross-grain checking or cracking and is very definite when the proper viewing angles are employed. There seems to be no difference in the type of checking from that met with in service. On veneer panels, cracking with the grain almost always appears early during the test preceding the strictly cross-grain cracks, and the time of appearance should always be noted and should form an important part of the rating of the lacquer in question.

It appears that the main problem is to develop standardized testing methods and equipment and enough experience and information in the intelligent application of such equipment.

Mr. R. J. Moore, Development Manager, Varnish Resin Division, Bakelite Corp., Bloomfield, N. J., said that, in reviewing the laboratory evaluation of paint and varnish films, the chemist is concerned with such fundamental factors as water resistance, flexibility and its retention, and resistance to chemical influences. Since the corrosion of metals or the deterioration of wood does not usually take place until water reaches its surface, the importance of water resistance is readily understood. In the laboratory this property is tested by immersion in cold or boiling water and examination of the film for defects developed during this exposure. Other measurements determining quantitatively the rate of penetration of moisture through the film have been proposed and may be preferable. The proper evaluation of the film qualities includes not only the determination of initial values but also their rate of change under exterior or accelerated exposure conditions.

The second factor, flexibility or distensibility, may be easily determined by a number of tests such as the Kauri reduction and mandrel bending tests. Again, while initial flexibility is important, more importance is attached to retention of this property on aging.

Chemical influences include oxidation, decomposition by ultraviolet light and radiations of longer wave length as well as the direct action of all those chemical agents encountered during use. The latter include soap and other alkalies, acids and the various corrosive gases

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[2] Wayne C. Norris, "Equipment for Testing the Resistance to Cold Checking of Lacquers and Other Surface Coatings," see p. 478.

present in the atmosphere. Alkali resistance is usually determined by immersion in weak sodium hydroxide solutions. Films containing relatively high concentrations of peroxide linkages are readily broken down under these conditions, while the films which dry by polymerization are admittedly more resistant. Resistance to other chemicals is frequently determined in much the same way by immersion in dilute solutions of the chemicals in question.

Mr. E. F. Hickson, Chief, Paint Section, National Bureau of Standards, Washington, D. C., discussed laboratory testing on inside flat wall finishes, especially as applied to the development of Government specifications. In the development of specifications based exclusively on physical and performance tests, considerable difficulty was encountered in devising tests which would give reliable numerical results. A large number of representative commercial paints were evaluated and specifications were based on the results shown by the best of them. Hiding power was determined by the usual brush-out test on black and white checkerboard, viewed under constant standardized illumination. Since reflectance greatly influences the hiding power of any given paint, a reflectance of not less than 85 per cent relative to magnesium oxide was specified. Although consistency is one of the most important paint properties, there is no commonly accepted procedure or instrument for the determination of this property. Brushing, flowing and leveling are determined by brush-out tests on sufficiently large beaver-board panels; small-scale tests, being unable to detect differences, are worthless for this purpose. Gloss is evaluated by comparison with a mutually agreed upon standard painted out at the same time and on the same surface as the paint under test. Flexibility is determined by a simple bending test of a baked finish on tin. The washability test consists of noting the ease of removal of writing ink and grease stains from dry paint films with grit soap and water. A specification based on these tests has resulted in an appreciable improvement of the quality of the paints submitted.

Topic II—Paints for Exterior Service on Wood:

Mr. H. A. Gardner, Chemical Engineer, The Institute of Paint and Varnish Research, Washington, D. C., introduced Topic II and summarized his experience with accelerated weathering tests on paints for exterior service on wood by stating that they had failed to give reliable results and that exterior exposure in Florida was much superior. Nevertheless, there was hope that more reliable testing methods and equipment could be developed and that this Symposium would stimulate efforts in this direction.

Mr. F. L. Browne, Senior Chemist, Forest Products Laboratory, Madison, Wis., expressed a somewhat similar viewpoint. In Mr. Browne's opinion, however, it is unreasonable to expect accelerated weathering to duplicate exactly in a series of paint coatings the same defects that appear over a longer period of time on exterior vertical south exposure. All that can reasonably be expected is that a weathering test will reveal in some recognizable way those inherent characteristics of coatings that we know from general experience affect their behavior under service conditions. It seems obvious that the radical change in the method of testing necessary to obtain the desired degree of acceleration should likewise seriously alter the manifestation of the visible defects. This defect of accelerated weathering may, however, be partly overcome by improved methods of interpretation. It appears unwise to discard accelerated weathering because of the present uncertainties in interpreting the results. Future development of accelerated weathering should concentrate not on some change in the cycles used but rather on a more careful study of the bearing which the defects of a paint as shown by accelerated weathering may have on the serviceableness of the same paint in actual use.

Mr. C. D. Holley, Director of Allied Research, The Sherwin-Williams Co., Chicago, Ill., ascribed many of the troubles of accelerated weathering to the lack of a well-established standard of normal weathering. The effect of variations in climatic conditions cannot be eliminated by the use of a guide paint on each panel. Although a few localities are known in which specific weather and climatic conditions repeat themselves in like cycles, performance under exposure in such localities would be badly misleading with respect to general service performance in other localities. Realizing the limitations of panel exposures, it would seem that definite information as to the average performance characteristics of the paint formulation in question could be obtained only if such exposures were repeated many times and under circumstances of application which satisfactorily simulate actual usage. With the aid of norms obtained in this manner, accelerated weathering tests could be standardized.

Mr. M. J. Dorcas, Manager, Lamp Dept., National Carbon Co., Inc., Cleveland, Ohio, on the other hand, expressed the opinion that even the most durable paints for exterior service on wood give typical failures in accelerated testing equipment if sufficient time is allowed, some of the best paints requiring from one to three months' continuous operation to get complete disintegration. If the light source contains more ultraviolet than sunshine, excessive chalking may be observed. More reliable results may, however, be obtained at the cost of increased exposure time. His experience led him to conclude that accelerated testing machines give a useful indication of the ultimate behavior of paints in actual service in a small fraction of the time required for the actual service test.

Mr. A. C. Eide, Sales Engineer, American Zinc Sales Co., Columbus, Ohio, pointed out that the attitude taken by technical men with regard to the value of the present test methods for weathering of paints depends in a large part on how much they demand of such a test. If perfect correlation with all kinds of paints is demanded, accelerated testing is a failure. However, the weatherometer is a splendid research tool although it is not yet developed for miscellaneous specification work. Experimental data indicate that the first appearance of failure in the weatherometer is a better guide than when the failure becomes more pronounced.

Mr. Harlan A. Depew, Research Chemist, American Zinc Sales Co., Columbus, Ohio, believes that what is needed is a test that in 24 hr. will give a good indication of the failure in 1 or 2 yr. of service. Based on the large amount of information gathered by the paint industry and a theory of the function of pigments in paint films, it should be possible to develop a truly accelerated test.

Mr. G. B. Girault, Paint Technologist, Robert W. Hunt Co., Chicago, Ill., reported very satisfactory correlation between weatherometer results and exterior exposure under service conditions. Both the degree and the nature of the breakdown obtained in accelerated weathering coincided satisfactorily with failure under exterior exposure.

Mr. C. Y. Hopkins, Division of Chemistry, National Research Council of Canada, Ottawa, Ont., Canada, conducted a series of tests in which the degree of correlation obtainable between accelerated weathering, 45-deg., and vertical roof exposures was determined. The test included a number of commercial and three experimental paints applied to white pine panels. The agreement between these three types of exposure was fair, although the weatherometer cycle used was found to be too wet. Where other observers have found discrepancies between natural and artificial weathering, it does not necessarily follow that the accelerated test is at fault. Unsatisfactory results from artificial weathering may in many cases be due to: (1) improper cycle in weathering machine, (2) inaccurate results from corresponding exterior exposures, (3) improper preparation of the panels, and (4) errors in interpreting the condition of the weathered film. It seems likely that when the most suitable conditions of test have been worked out, the accelerated weathering test will be found to be as reliable as, or perhaps more reliable than, outdoor weathering.

Mr. E. W. McMullen, Director of Research, The Eagle-Picher Lead Co., Joplin, Mo., reported very poor correlation and contradictory results between accelerated weathering and exterior exposure in a series of tests conducted in an accelerated weathering machine of their own construction. Believing that improved sources of light, more closely simulating sunlight, would yield more satisfactory correlation between accelerated and exterior exposure results, he is making attempts to design an improved accelerated weathering machine. Although final results are not yet available, there are indications that better agreement will be secured. In the meantime, vertical exterior exposures are used almost exclusively in the evaluation of paints.

Mr. C. H. Rose, Chemist, National Lead Co., Research Laboratories, Brooklyn, N. Y., also reported poor agreement between accelerated and exterior weathering in regard to type of failure and life expectancy. The accelerated weathering system used by this investigator has in many cases developed checking in paints to a much greater degree and proportionately earlier than exterior exposure. It also caused cracking and scaling in paints usually not subject to these defects under normal exterior weathering. On the other hand, it has been observed that accelerated weathering often does not develop chalking to the degree which is obtained on exterior exposure. However, accelerated weathering has proven valuable as a research tool, especially in the elimination of paints having poor weathering characteristics. It has also been used quite successfully in the evaluation of the color stability of tinted paints, although the type of fading caused by change in refractive index relations as the vehicle disintegrates cannot be determined in this way, because this involves the breakdown of the film which is not accurately reproduced by this machine. The poor correlation between accelerated and exterior weathering may be attributed to the fact that in accelerated weathering the concentrated weathering influences are imposed on young and immature films while under normal exterior exposure the much milder and less concentrated weathering conditions are spread over a much longer period of time. During much of the weathering period outdoors, the films are fully matured and are affected by the elements in a very different way. It might be possible to obtain accelerated weathering results which would correlate somewhat better with exterior exposure by using different types of cycles for the various types of paints, or even for the various types of failures.

Mr. S. Werthan, Chief, Paint Section, Research Division, The New Jersey Zinc Co., Palmerton, Pa., is of the opinion that accelerated weathering systems should be so formulated as to reproduce the average failure characteristics of a number of standard paints carefully established by a large number of exterior exposures. In a balanced accelerated weathering test, the factors promoting disintegration of the paint film under exposure and the frequency of their occurrence are generally intensified, while their correct relation and sequence are maintained. Since these factors affect various types of finishes to different degrees, an accelerated exposure cycle should be developed for the specific finish to be tested. Usually an artificial cycle will not exactly reproduce the deterioration occurring under exterior exposure, and consideration must be given to the type of exposure and the interpretation of the results. Absolute failure ratings cannot be given and the durability of a paint should be expressed in terms of a standard comparison paint of known durability and failure characteristics. In general, good agreement is obtained between accelerated and exterior exposure results as far as the best and the poorest paint of a series are concerned. Lack of agreement is with the intermediate products of a series, which usually differ only slightly in durability characteristics. Many years of experience with accelerated weathering tests justify the conclusion that accelerated weathering provides reliable information if: (1) the apparatus and cycle are properly designed and calibrated, (2) the panel preparation is carefully standardized, and (3) the failures are accurately observed and interpreted.

Topic III—Paints for Iron and Steel:

Mr. C. F. Rassweiler, Director, Philadelphia Laboratory, Finishes Division, E. I. du Pont de Nemours and Co., Inc., Philadelphia, Pa., introduced Topic III and called attention to the fact that the broadness of the field covered prevented a universal application of any one accelerated test. Conditions of exposure vary so widely that accelerated tests which might correlate well in one case are likely to fail completely in other cases. It is, therefore, well to keep in mind whether the finish to be tested is, for example, the enamel type as used on automobiles, or the maintenance type as used on structures.

Accelerated tests may be broadly classified into: (1) tests which attempt to duplicate actual service, (2) tests which depend on the concentration of some single or perhaps relatively small group of destructive influences, and (3) tests which attempt to predict durability on the basis of certain fundamental film properties such as flexibility, water impedance or abrasion resistance. In selecting accelerated tests in any of these three groupings, care should be taken to determine whether the results obtained are reproducible with the desired accuracy, whether the failure produced is related to the failure observed under actual service conditions, whether the test places the materials in question in the proper order of relative durability, whether the results indicate the relative length of time before failure may be expected in service, and whether the range of compositions over which this test yields reliable correlation with actual service has been definitely established. Much of the confusion in accelerated testing has probably been due to attempts to push accelerated tests beyond the range on which correlation had been established.

In considering the correlation between accelerated testing and actual service durability, it is only fair to point out that the base against which correlation is being attempted, namely, service durability, is itself not a constant or easily determinable value. Even with a relatively large number of units available for testing, it is extremely difficult to eliminate accidental differences in exposure conditions and to establish any definite value as representing the service durability of a given finish. Test fence exposures usually relied upon as comparison standards are in themselves something of an accelerated test greatly affected by local conditions, the method of preparing the specimen and other factors, many of which are not fully understood. It might well prove that the consensus of opinion expressed in this Symposium will be to consider the problem of standards before much study is given to concentrating the weathering factors themselves.

Mr. G. B. Girault, Paint Technologist, Robert W. Hunt Co., Chicago, Ill., followed and stated that the results obtained in extensive work on the inspection and testing of metal protective paints for the Navy Department, railroads, highway departments, etc., had demonstrated the dependability of accelerated tests as compared with actual outside durability, citing examples such as a license plate finish, a paint for use on steel railroad cars and a gasoline truck enamel. The durability of these finishes as determined in the weatherometer was verified by the results of actual service.

Mr. S. G. Saunders, Staff Officer in Charge of Paint Methods Dept., Chrysler Corp., Dodge Brothers Division, Detroit, Mich. expressed a similar viewpoint. After extended experiments with a machine using a quartz-mercury arc which yielded very discouraging results, a machine using a carbon arc which has been in use for a period of two years has yielded failures very similar in design and pattern to the failures observed under exterior exposure in Florida. The exposures were carried to the point of failure by checking, cracking, peeling, blistering, alligatoring, etc. As far as chalking is concerned, it has not been possible to correlate between accelerated tests and actual service. However, it seems possible that by changing the composition of the carbons and by interposing a Corex filter, results might be produced which would be more in line with those obtained in actual service. In the case of lacquers, the correlation between Florida and accelerated exposures amounted to 75 per cent, whereas there was about 90 per cent correlation in the case of synthetic resin enamels. The type of resin used, undercoater system, and variations in formulation account to some extent for the differences observed. In the case of red colors, both lacquers and synthetic resin enamels show no similarity between service and accelerated exposures. Taking all classes of materials into account, about 10 to 12 hr. accelerated exposure are equivalent to approximately 1 month's exposure in Florida.

Mr. V. M. Darsey, Technical Director, Parker Rust-Proof Co., Detroit, Mich., limited his discussion entirely to his experience with the salt-spray test. He concludes that the salt-spray test has proved to be a convenient and practical method of estimating the comparative durability of most paint coatings applied over ferrous surfaces. The test is particularly valuable for the determination of the useful life of exterior finishes, in which corrosion is the primary factor. In general, those coatings which show the least amount of breakdown in the salt-spray test have been found to be most durable by exterior exposure tests. Much criticism of the salt-spray test results is due to attempts to establish a definite comparison between a given period in the salt spray and the equivalent period of months outdoors. The salt-spray test data indicate primarily the effect of corrosion on the durability of the paint coating, and such factors as variability in outdoor temperature, effect of sun, fading, and chalking are not within the scope of the test. Although the salt-spray test simulates marine atmosphere it is nevertheless an artificial condition and it is believed undesirable to estimate the actual service life of a paint coating on the basis of salt-spray results. In the grading of failures on panels subjected to the salt-spray test, it has been found very useful to express the amount of rusting observed by the distance rust crept underneath the paint film from a scratch made in the film with a sharp point prior to exposure. Consideration should be given to this method for making the results obtained with this test more reproducible.

Mr. E. H. Barlow, Chief Engineer, Standard Oil Development Co., Elizabeth, N. J., summarized the status of accelerated testing of metal protective paints in the petroleum industry. For 18 yr. the large oil companies have been conducting accelerated as well as service tests and have come to the conclusion that the latter are more reliable and give more complete information regarding the product under test. Accelerated tests serve a purpose in so far as they give some information in a much shorter time, facilitate the testing of a much larger number of products than would be possible otherwise, help eliminate unsuitable coatings, and afford a more intelligent selection for service tests and future use. Accelerated tests should not be too severe, but should simulate as closely as possible, the conditions encountered under actual service. One useful application of accelerated weathering machines such as the weatherometer is the evaluation of the tint and gloss retention of color enamels for pumps, tanks, trucks and other equipment. In the evaluation of finishes for the interior of gasoline tanks, an exposure cycle consisting of exposure to salt water, moist air, hot water, and cleaning compounds has been found more reliable than accelerated weathering tests of the conventional type. For other special conditions, preference is given to tests conducted under actual service conditions, especially where those do not require a long time before failure is observed.

Mr. C. E. Wilson, Technical Director, James B. Sipe and Co., South Hills Branch, Pittsburgh, Pa., reports poor correlation between accelerated tests and exterior 45-deg. exposure. Of twenty series of exposures, eight showed good correlation in the order of failure, ten showed no agreement and two partial agreement. Those tests which showed good correlation included five series in which the accelerated cycle was relatively short and the test fence exposure relatively long. They also included three sets in which both accelerated and exterior exposures were long. Several series of tests have been carried through the accelerated weathering cycle for very long periods without the development of the type of failure which occurs on the test fence. After chalking is well started, it apparently protects the remainder of the coating and several tests were observed which retained their protective value much longer in the accelerated weathering machine than under exterior exposure. One exception to the general unreliability of this accelerated weathering system was found in the testing of clear varnish films over bare metal. It is this investigator's opinion, therefore, that accelerated weathering is useless except for a comparison of the resistance to fading of pigmented films, and for a comparison of the durability of unpigmented varnishes. The results of those tests, however, should be corroborated by test-fence exposures.

Messrs. J. C. Moore and N. T. Phelps, Superintendent, and Chief Chemist, respectively, Paint Dept., Sinclair Refining Co., Marcus Hook, Pa., also found accelerated weathering to show no correlation so far as rate and type of failure are concerned with the same failures on test-fence exposure. The weatherometer is unsatisfactory for evaluating the anticorrosive properties of primers and for testing aluminum paints, although there is fair correlation between accelerated and test-fence results for oleo-resinous varnishes and enamels. The weatherometer was found most valuable for comparison of gloss retention and checking and cracking failures, but is believed to show unreliable results in the case of tint retention, water resistance and water spotting. Testing marine finishes by exposing panels on a rack on an oil tanker has been found a more satisfactory method than the use of accelerated weathering. Accelerated weathering is no doubt valuable as a research tool but cannot be used with satisfactory results by the ordinary person. In the hands of the experienced operator, the weatherometer may be used to secure very satisfactory results, while in the hands of the untrained or unscrupulous user, the results of the weatherometer may be practically worthless.

Mr. H. A. Nelson, the chairman, in concluding this Symposium, stated that although many of the statements heard appear to be conflicting and impossible of reconciliation, the more or less general impression is justified that accelerated laboratory tests even in their present elementary stages, have proved very useful research tools which cannot be ignored, although their application to specification purposes should still remain quite limited.

EQUIPMENT FOR TESTING THE RESISTANCE TO COLD CHECKING OF LACQUERS AND OTHER SURFACE COATINGS

By Wayne C. Norris[1]

The impelling force most responsible for the change from varnish to lacquer a few years ago was the demand in the consuming industries for greater speed of production. As we all know, the general adoption of nitrocellulose lacquer reduced finishing schedules to a fraction of the time formerly required. It may be said that lacquers were the result of the substitution of properly plasticized nitrocellulose for most or all of the oil and part of the resin in the oleoresinous materials which they replaced. No compromise with protection was necessary, even though the schedules were so enormously speeded up.

Once, however, the initial hurdle of eliminating days from the finishing schedule was jumped and production lines made their appearance, it became desirable further to reduce time schedules as far as safety would permit. Much of this further speeding up necessarily had to be done by the development of resins which combined maximum toughness with the desired working properties, such as fast dry and solvent release, good sanding, rubbing, polishing, etc., which properties have always been accompanied to a greater or less degree with lack of distensibility or "shortness." Kauri-reduction tests, bend tests, accelerated exposures, various distensibility measurements on the films themselves, and exterior exposures have all been used by the paint and allied materials manufacturers in their efforts to determine the safe limit in "shortness." The testing of finishes for their resistance to cold checking (tendency to rupture under severe temperature change) has until recently been done only sporadically and is not yet a common routine test. Its importance, however, especially in the furniture field, is attested by the fact that in evaluating finishing materials, one leading consumer places greater stress on "cold check resistance," than on any other property, such as print resistance, or application and rubbing qualities and resistance to heat, moisture and solvents. It is at once a test of tremendous practical significance because finishes in use are subjected to sudden temperature differentials, and is of fundamental importance because it yields results not entirely predictable by other accepted tests. In the sense that print resistance is a measure in one direction of the useful thermo-plasticity range of a coating, resistance to cold checking is a yardstick at the other end of the range.

Notwithstanding the fundamental nature and importance of such a test, there is need for agreement in the industry as to how it should be carried out. Consistent data in a single laboratory have been difficult to obtain while check results between two or more laboratories have been almost impossible.

While a test so dependent upon other

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[1] Resin Development Laboratory, American Cyanamid and Chemical Corp., New York City.

factors, such as application, might never be expected to yield highly quantitative results, the main difficulty has been the lack of a suitable and standardized apparatus in which to test.

A survey of the methods employed for such tests in a number of lacquer laboratories showed considerable variation, ranging from the use of the mechanical water-cooler in the office, to utilizing a refrigerated ware-house. An average method was the adaptation of a rather large-size mechanical refrigerator, but this provided no adequate control. The capacity was insufficient for cooling a set of panels in the desired time and, without air circulation, temperature differentials in the compartment of 10 to 20 deg. Cent. were commonly experienced.

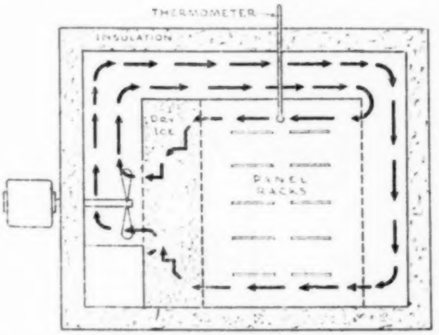

Fig. 1.—Insulated Electric Oven Packed with Dry Ice.

Preliminary Experiments:

The stringent requirements to be met and the lack of adequate refrigerating equipment to simulate service conditions suggested the use of dry ice in a cabinet as the cooling medium. Accordingly, its possibilities were examined in a well-insulated electric oven of the induced draft recirculating type. It was found that with about 40 lb. of dry ice loaded into a wire cage and placed in the oven as indicated in Fig. 1, temperatures of from −20 to −40 C. could be reached in 15 to 20 min. after placing the panels, previously warmed to 49 C., in the compartment. With no more control than is possible in a setup of this kind, however, duplication of results is difficult. Standard samples must be run with every series, and results expressed in terms of the behavior of those standard samples. Notwithstanding the obvious faults of such a setup, the equipment is available in most lacquer laboratories and it represents an economical means by which cold check tests may be made and can be recommended for those laboratories with only an occasional cold-check test to run.

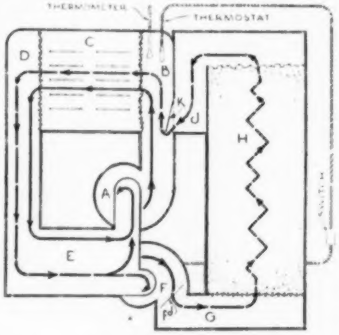

Fig. 2.—Flow Chart of Cabinet.

Description of Apparatus:

Once the value of solid CO_2 as a cooling medium was demonstrated, a cabinet was designed to furnish as economically as possible sufficient capacity to lower the temperature of 50 lb. of steel panels from 120 F. (49 C.) to −5 F. (−20.5 C.) in 5 min.

A flow chart of the design is shown in Fig. 2. Briefly, the apparatus consists of an insulated cabinet containing a panel compartment, two dry-ice compartments, a continuously operated circulatory system, and an intermittent cooling circuit. In the flow chart, the solid arrows represent the continuously operated system. Here the gases pass from fan *A* through duct *B* into panel compartment *C*, and then back through duct *D* and *E* into fan *A*. The secondary or cooling circuit is shown by broken arrows and is cut into operation through the thermostat. The gases are taken from the common duct *E* through fan *F* and the manually operated damper *fd* into the chamber *G*. From here they are cooled by passing up through the dry-ice compartment *H* and are then allowed to escape from duct *J* through rubber check valve *K* into duct *B*, where they mix with the gases in the main circulating system.

Description of Cabinet:

The cut-away side elevation shown in Fig. 3 is taken looking across the broad side of both dry-ice compartments. That is, the dry ice compartment *H* covers the whole of the area directly in front of the observer, while the compartment H_1 covers a similar space in back of the fans, panel compartment, etc. The solid arrows in Fig. 3 show the flow of the gases in the main circulatory system which is in continuous operation, while the intermittently operated cooling circuit is shown by broken arrows.

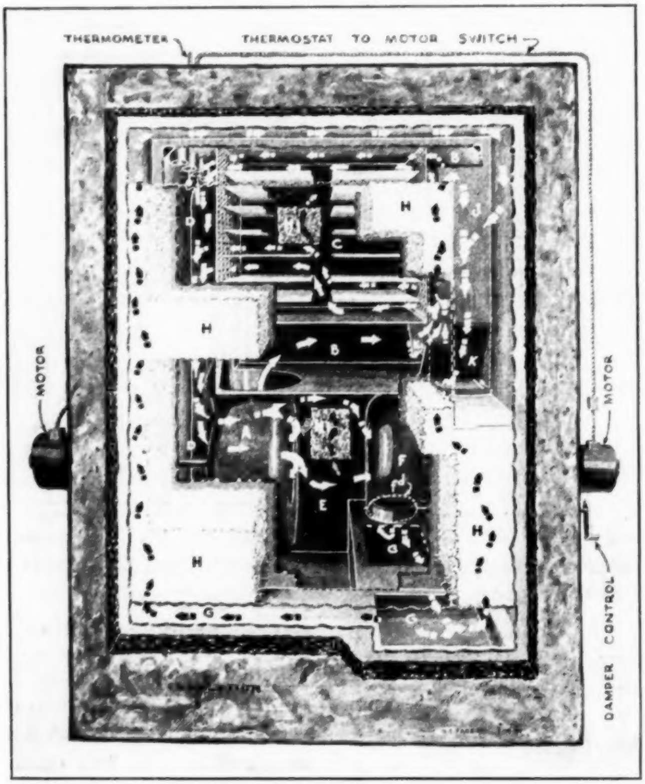

FIG. 3.—Test Cabinet.

Specifications:

The details of the apparatus are as follows:

OVER-ALL DIMENSIONS: 2 ft., 10 in. by 2 ft., 4½ in. by 4 ft.

COMPARTMENT DIMENSIONS: 1 ft., 1 in. by 1 ft., 3 in. by 1 ft., 1 in.

CONSTRUCTION: $\frac{3}{16}$-in. steel box with galvanized sheet steel ducts and the screened openings covered with tinned 8-mesh wire. Cabinet carried on four swivel mounted castors with 4-in. rubber tired wheels.

INSULATION: 3-in. cork insulation over entire outside surface, covered with 28-gage steel.

CIRCULATORY SYSTEM:

Motor.—$\frac{1}{4}$ h.p., 1800 r.p.m. totally enclosed vertical flange mounted ball bearing.

220 v., single phase, 60 cycle.

Fan.—Buffalo Forge No. 3 baby conoidal.

COOLING SYSTEM:

Motor.—Same as for circulatory system.

Fan.—Buffalo Forge No. 21 reversible volume fan.

THERMOSTAT: Liquid filled bulb, mercoid type. Range −6.7 C. (+20 F.) to −34.4 C. (−30 F.).

Differential 2 to 4 deg. Fahr.

PANEL RACKS: Two: each accommodating 15 panels 6 by 12 in. with supports $\frac{11}{16}$ in. apart.

Operating Characteristics:

Damper Manipulation.—The main function of the damper is to assist the thermostatic control by regulating the amount of cold CO_2 gas allowed to enter the main circulating system when the intermittent fan is in operation. It also controls the amount of back draft or feed into the main circulatory system when the intermittent fan is not operating. In general, it has been found that the damper should be kept closed or nearly closed when working around −10 C. (14 F.) and wide open at −35 C. (−31 F.). At a working temperature of −20 C. (−4 F.), it will be found advantageous to open wide the damper when cooling down the box and during the first cooling period after the panels are introduced. After the initial drop is accomplished and the thermostat reset, the damper should be closed for a while until equilibrium is established, when it may be opened up at least partially.

Circulation.—Anemometer readings were made by placing the instrument in the center of the panel compartment. The following data were obtained by running the cabinet without solid CO_2 being charged into the compartments:

		FT. PER MIN.
Both fans on	damper open	480
Circulating fan only	damper open	380
Intermittent fan only	damper open	100
Circulating fan only	damper closed	370
Intermittent fan only	damper closed	0

These results show a degree of circulation which permits rapid heat transfer and also show the positive action of the damper.

Typical Cycle.—Starting with the cabinet at room temperature and a charge of 80 lb. of dry ice (approximately 40 lb. in each compartment), the temperature of the gases coming across the working chamber will stabilize at −20 C. (−4 F.) in 30 to 40 min. with the thermostat set for that temperature. If that is the temperature at which it is wished to cool the test panels, the cabinet should be cooled to about −26 C. (−15 F.). With both motors shut off, the panels previously heated to 49 C. (120 F.) in a separate oven, are introduced, the motor switches are then closed, and the thermostat reset for the required temperature. With this procedure the temperature of the gases will stabilize at −20 C. (−4 F.) within 5 min. with the compartment filled to its capacity load (two aluminum racks containing 15 veneer panels each, with panels and racks introduced at a temperature of 49 C. (120 F.).

Working Range and Economy.—Temperatures within the range of the thermostat (−6.7 to −34.4 C. (20 to −30 F.)) can easily be reached in 5 to 6 min. and held within ± 2 deg. Cent.

when the general procedure given in the above paragraph is followed. The quantity of dry ice charged may vary from 30 to 120 lb., depending on the temperatures required from −10 to −35 C. This observation is made not only from the point of view of economy, but also because of the better control possible.

A charge of 80 lb. of dry ice is sufficient for one day's continuous operation at a temperature of −20 C. This is allowing for a change at the end of each of the 8 hr. If two sets of panels are being alternated between heat and cold, this means that 60 panels may be given four temperature changes per working day of 9 hr. At a cost of 2½ cents per pound of dry ice, this amounts to $2.00 per day.

Uses:

Most of the testing to date with this apparatus has been on furniture lacquers applied to wood veneer panels. It is equally as useful, of course, in testing other types of finishes, for automotive or airplane use, for example. Physical tests, such as bending, may be made on panels while they are in the compartment after they have been cooled. The circulating fan should, of course, be cut off at all times while the lid is removed. By opening from the top and employing CO_2 as the cooling gas, relatively little heat interchange is experienced during the short time required to make bending tests.

The cabinet has also been found to be especially useful in the cooling of liquid samples. The compartment is sufficiently large to permit filtration and some other operations.

Cold Checking—General Considerations:

The thermal coefficient of linear expansion for wood is considerably greater across grain than it is with the grain. It ranges from five times as much for chestnut to twenty-three times for beech, with the value for maple and walnut at approximately seven and one-half times. As the coefficient of expansion for lacquer is high and more nearly like that of wood across the grain, rather than with the grain, films could be expected to tend to follow the wood in its movement across grain and thus show relatively little tendency to crack with the grain. Conversely, the lacquer film would tend to expand or contract to a greater degree than the wood in the direction with the grain, and thus set up relieving cracks perpendicular to this movement, which amounts to cross-grain checking. In practice, however, the failure is not so definitely and predominantly of this type as one would like to expect. This is due to such interfering factors as lack of perfect film continuity, which probably favors cracking with the grain, and to the fact that when the panel is subjected to the cold the film is chilled—and probably thoroughly chilled—before the wood undergoes appreciable cooling accompanied with its unequal contraction. Under these conditions insufficiently plasticized lacquer would, therefore, tend to immediate rupture equally in all directions, or in view of the above mentioned film continuity effect, mostly in a direction with the grain. On the other hand, if the film remains intact until the wood reaches the same temperature, the differentials again become operative and cross-grain failure is favored. Experience has shown that cross-grain cracking or checking can properly be taken as the end point. It may be associated with a greater or less amount of cracking with the grain and is often transverse or partially of each type.

When examining a panel for cold checking—that is checking or cracking not with the grain—all other types of cracking and film imperfections can be

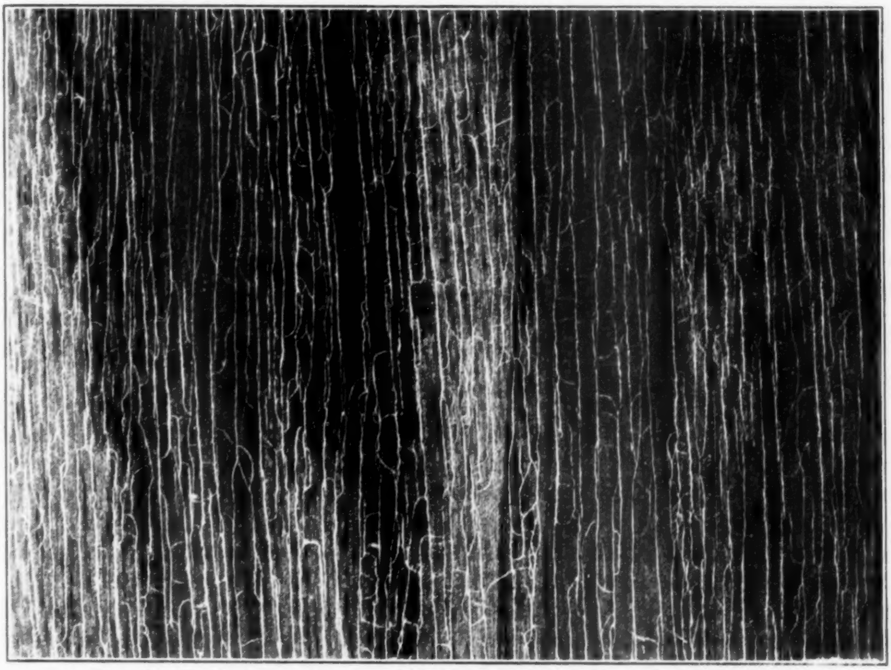

FIG. 4.—Typical Fareuil of a Wood-Finishing Lacquer by Cold Checking.

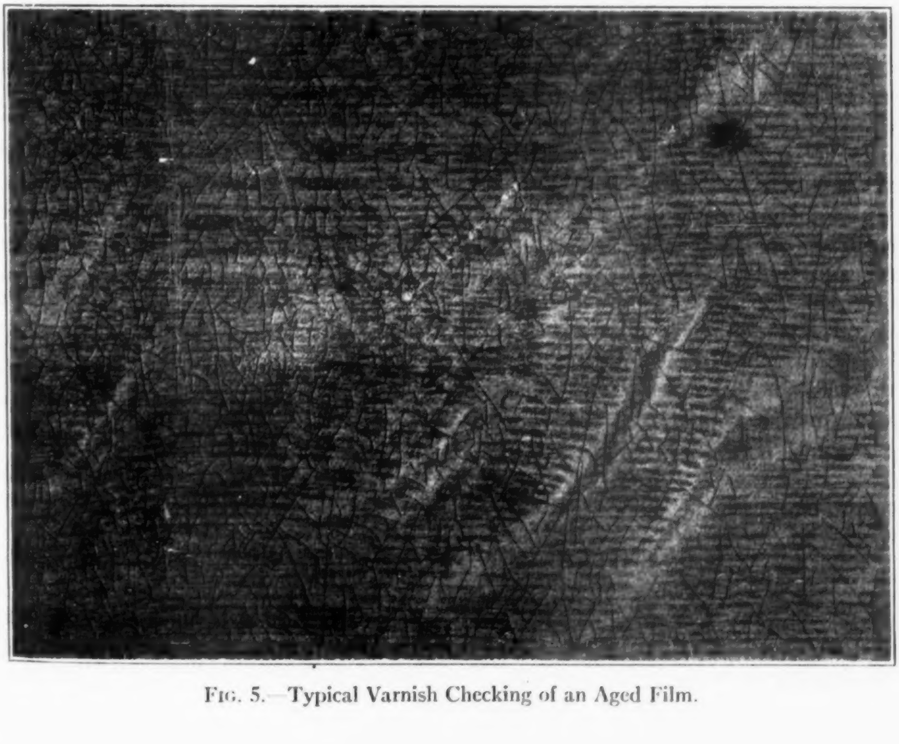

FIG. 5.—Typical Varnish Checking of an Aged Film.

easily eliminated by the use of proper illumination and viewing angle. The observer should stand facing a dark background and observe the panel at an acute angle with the light source at his back. It is much easier to observe than to photograph because of the small field and long depth of focus necessary in a proper representation. Neither viewing nor lighting angles need be so acute if the failure has proceeded farther than that just necessary for visual detection.

Figure 4 is a typical failure of a wood-finishing lacquer by cold checking which has been carried far enough to permit photographing. Figure 5 shows the typical varnish checking of an aged film under service conditions. Photographic representation is here facilitated because the size and permanence of the cracks has permitted dark dirt to become imbedded and thus contrast the design against the light oak base.

Typical Results:

As typical of tests made with this machine the following comparisons are drawn between three lacquer formulas which were tested over walnut veneer panels which had been stained, coated with shellac, filled and sealed under identical schedules. The sealer in each case consisted of 100 parts ½-sec. nitrocellulose, 100 parts Teglac Z-152, 50 parts Rezyl 387, and 20 parts each of zinc stearate and dibutyl phthalate with the customary solvents. The formulas of the three clear lacquers are as follows:

	LACQUER No. 1	LACQUER No. 2	LACQUER No. 3
(Dry) ½ sec. nitrocellulose.	100	100	100
Teglac Z-152 (hard friable resin)...........	50	100	200
Rezyl 387 (semi-plastic resin)................	50	50.	50
Dibutyl phthalate......	25	20	20
Blown castor oil.........	25	20	...

Teglac Z-152 used in the above lacquer formulas is a rosin modified alkyd resin of an extremely hard and brittle type. Rezyl 387 is an oil modified alkyd resin which is considerably softer and more plastic than the Teglac type. Marked differences in results are obtained by varying the proportions of these resins and the proportions of total resin to nitrocellulose and plasticizer in the finish.

Lacquer No. 1 was run for 18 cycles and showed no failure. It can probably be considered as of the excessively safe type.

Lacquer No. 2 showed very slight failure at the ninth cycle and is of the intermediate type.

Lacquer No. 3 showed definite failure on the first cycle and, therefore, is too brittle to meet very rigorous requirements of the cold check test.

Some laboratories have considered the use of solid maple panels in testing finishing lacquers because of the better uniformity it is possible to obtain. Our results indicate too high a degree of perfection for such surfaces. Lacquer No. 2, for example, was still in good condition after 39 cycles on a 6 by 12 by ½-in. maple panel.

Glass and steel have also been tried as time-saving substitutes for wooden panels. Plate glass appears to have some possibilities, but the work is not sufficiently far along to warrant its recommendation.

In conclusion, it is hoped that this paper will stimulate discussion of the factors entering into the testing of finishes for their resistance to cold checking.

Acknowledgment.—Much credit is due the members of this organization under the direction of Dr. A. O. Jaeger for their helpful cooperation, especially the Engineering Department under the supervision of its chief engineer, J. E. Jewett.

A LABORATORY CHANNEL TEST FOR GEAR OILS

By J. P. Stewart[1]

Synopsis

One very important characteristic of a gear lubricant is the channeling temperature, yet no laboratory test is available for its evaluation. The pour point has been used by some for this purpose and a number of special tests have been suggested, yet industry at large has had to depend upon service experience or upon tests in full-size axles.

The first prerequisite of a laboratory channel test is correlation with oil performance in axles. Therefore, axle channel tests at low temperatures formed the first step in the development of such a test. In the axle tests the channeling temperature was taken as that temperature at which 40 per cent of the oil was in circulation after 10 min. of axle operation.

Channeling was found to be most severe at the lower speeds and most of the work was done at an axle speed equivalent to 20 m.p.h. car speed. It was found also that the temperature reached during operation had a profound effect upon distribution after rechilling, due to an air-oil emulsion which persisted during the rechilling period. Under such conditions channel temperatures varied between 20 deg. Fahr. below and 20 deg. Fahr. above the A.S.T.M. pour point.

On the basis of these axle data a laboratory test was developed which gave reasonable correlation with the observed performance of the oils in the full-size axles. The test employs standard A.S.T.M. pour-point equipment with the addition of a special stirring apparatus which reproduces the aerating action of the rotating axle gears. While the procedure is somewhat more elaborate than the regular A.S.T.M. pour-point determination, it is not difficult of execution and is as reproducible as that test. It would appear that the method has been developed to the point where it can be given consideration by the A.S.T.M. as a tentative channel point test.

The Society of Automotive Engineers' handbook under "Recommended Practice" for gear oils carries only two specifications: one, viscosity; two, the notation "Must not channel in service at deg. Fahr.," followed by specific temperatures for the several grades. Even the layman knows that below some nebulous temperature a lubricant will not function properly because it becomes "too thick"; all of us have been confronted with the question of the minimum temperature at which a gear lubricant can be used before failure of lubrication results from channeling. Yet in the face of this generally acknowledged need, no laboratory test is specified or available by which the channeling point of a lubricant can be determined.

One obvious way to evaluate this

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characteristic is to test the lubricant in an axle, but this, at best, is a clumsy and time-consuming undertaking and if a cold room is not available even that method is denied us. Efforts have been made to use the A.S.T.M. pour point[2] of the oil to supply this information, even though experience has shown that this test does not give a true indication of the temperature at which channeling can be anticipated in service. Other tests have been suggested but none seems to have stood the test of time, at least there are none apparently correlated sufficiently with actual performance data to warrant unqualified acceptance. It was because of this need that the development of a simple reliable laboratory test was undertaken.

Since the first prerequisite of a test of this nature is that it be based upon and correlated with actual performance of oils in axles the work was started with tests in two axles in a low-temperature room with a third axle added later. The test oils, Table II, wese quite varied as to source, pour point, and viscosity and represented the range of oils usually used in axle lubrication.

Axles were employed rather than transmissions because experience has shown that channeling will occur at a higher temperature in the former than in the latter. The obvious explanation of this is the greater number of gears in the transmission and their relatively small separation which produces a greater degree of oil churning in that unit than in the axle.

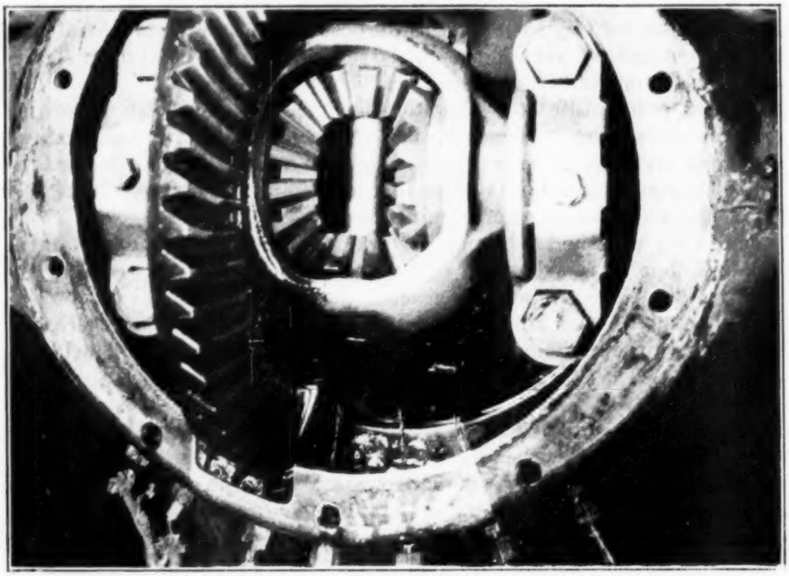

FIG. 1.—Differential Housing Showing Method of Installing Thermocouples to Indicate Circulation of the Oil.

One of the chief drawbacks with oil-distribution studies in axles lies in the fact that the observer can see only the surface of the lubricant regardless of the number of peepholes provided in the housing. Such observations do not produce very satisfactory test data.

X-ray spectacles seemed to be de-

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[2] Standard Method of Test for Cloud and Pour Point (D 97 – 34), 1936 Book of A.S.T.M. Standards, Part II, p. 853.

manded. However, while it was impossible actually to see movement below the surface, temperature changes which accompany such movements could be observed by judicially placed thermocouples. For very rapid movement at low temperatures the rise in temperature can be as rapid as 35 deg. Fahr. a minute. Accordingly, thirteen thermocouples were placed in an axle housing at different depths in the vertical plane of the axle shafts, as illustrated in Fig. 1. The specific locations are indicated in Fig. 3. The thermocouples were made of No. 24 B. & S. gage wire and were bound to piano wire standards with thread; the standards were mounted into fittings which were screwed through the bottom of the axle housing. The bulk of the couples and standards was kept small so that the couples would not interfere with the movement of the lubricant. *An additional thermocouple* placed in the pinion housing at the forward pinion bearing and a small drain plug placed in the same general location provided a means of determining the inception of oil supply to that bearing.

In addition to these subterraneous indications of oil movement the usual "peepholes" were placed in the axle housing above the oil level, through which the observer could obtain a clear view of surface effects within the axle.

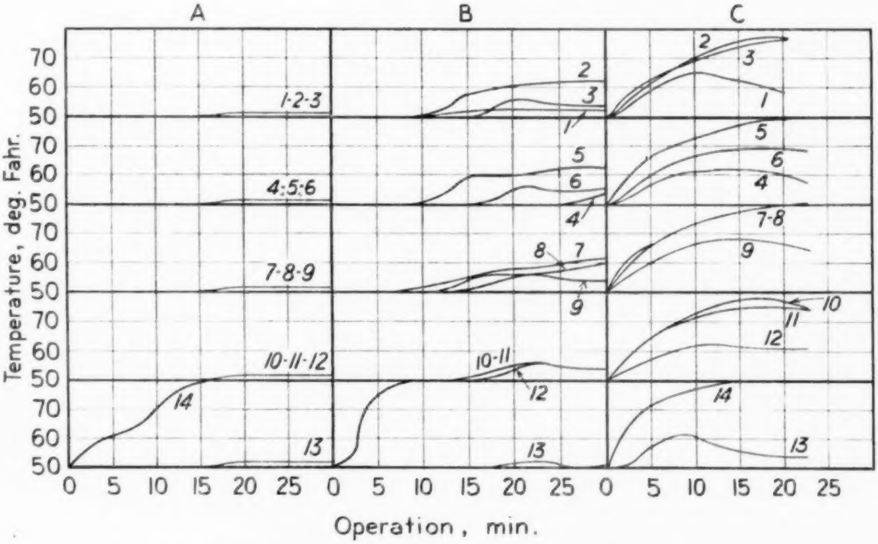

FIG. 2. – Graphic Log of Temperature Gradients of Axle Thermocouples.

Marked break in temperature gradient indicates oil movement at thermocouple location. Thermocouples numbered to correspond with locations indicated in Fig. 3.

Conditions of Test:

(*A*) Circulation at 50 F. with 70 F. prechilling temperature.
(*B*) Circulation at 50 F. with 100 F. prechilling temperature.
(*C*) Circulation at 50 F. with 125 F. prechilling temperature.

Lubricant II

The axle was mounted in a cold room and driven by a dynamometer outside the room. Axle temperature control was effected by the cold room air temperature with a natural rate of cooling (not accelerated by a blower). This naturally prolonged the work to some extent but its advantage lies in the reproduction of the chilling rate experienced in actual service, an important feature when dealing with certain types of oils.

higher axle speeds. Thus the data obtained under the lower speed condition apply not only to those cars which are operated mainly at the lower speeds but to practically all cars during the winter season.

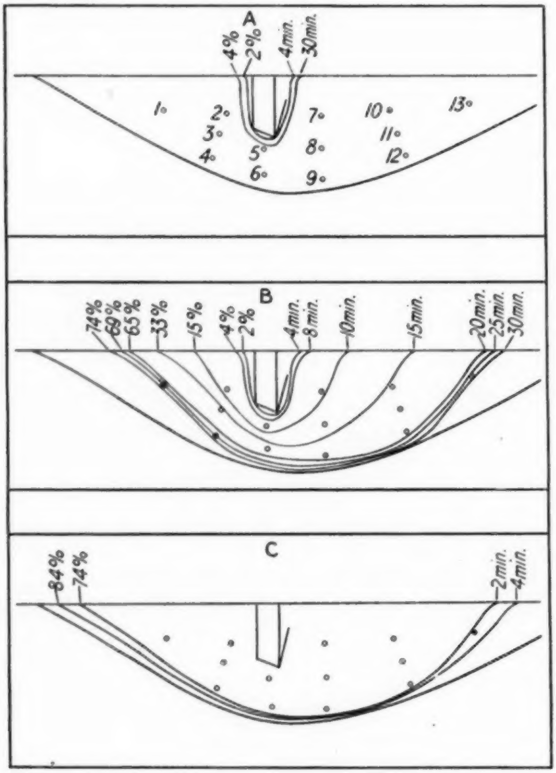

FIG. 3.—Axle Circulation Contour Curves Plotted from Graphic Log of Fig. 2.

Figures denote time intervals and percentage of oil in circulation. Thermocouple identifying numbers indicated in (*A*).

AXLE TEST PROCEDURE

Previous experience plus a number of trial runs indicated that the most severe channeling occurred at the lower speeds. For that reason 1000 r.p.m. pinion speed (20 m.p.h. car speed) was selected for this work. The more severe channeling at this speed is doubly significant since it was found that a condition of channel can be set up which persists even at

The actual test procedure was rather simple. The thoroughly cleaned and dried axle was charged with a test lubricant to the normal filling level and operated upon a prechilling and distribution run to a predetermined oil temperature, after which the room and axle were chilled to the test temperature, the first run on each oil being made at the pour point. Upon subsequent tests with the same charge of oil, the prechilling run was a prolongation of the test run, which was continued until the oil had reached the selected temperature, external heat being used if necessary. The prechilling run was repeated before each low-temperature run. From the inception of each channeling run, the temperatures registered by the 14 thermocouples were read as rapidly as possible ($2\frac{1}{2}$ min. for a complete cycle). Continuous observation was also made through the peepholes in the axle housing, of the circulation of the oil to the different axle parts.

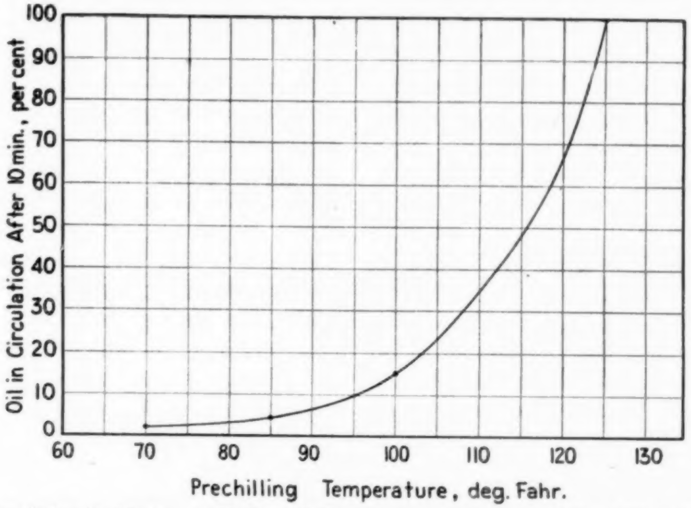

FIG. 4.—Circulation in 10 min. at 50 F. Oil Temperature with Several Prechilling Temperatures (Lubricant H).

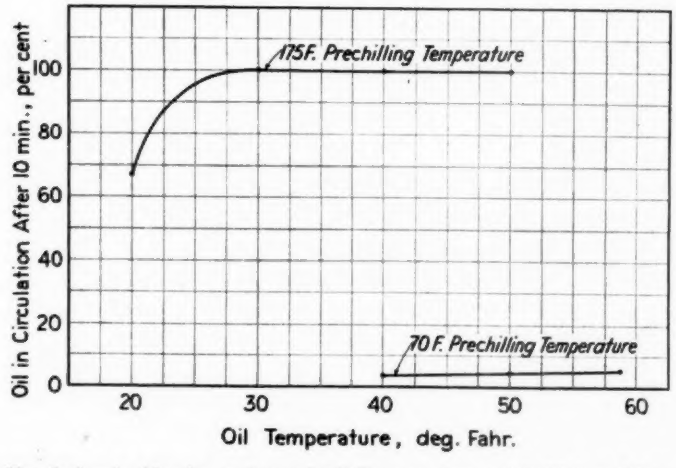

FIG. 5.—Circulation in 10 min. at Several Oil Temperatures with High and Low Prechilling Temperatures.

The temperature data so obtained were placed in graphic log form and the time interval observed at which the temperature gradient for each thermocouple showed a distinct change. This break in temperature gradient was taken as an indication of oil movement, that is the establishment of circulation at that point. Specimen graphic log sheets are illustrated in Fig. 2 showing, respectively, distinct channel, insufficient distribution, and essentially immediate 100 per cent distribution.

From the above data, boundary circulation contours were drawn on a vertical section through the axle showing the cross-sectional area of the oil in motion after stated time intervals. Figure 3 shows such plots taken from the data of Fig. 2. The percentage of oil in circulation at any given time interval was indicated by the relative areas of the total oil body and that within the boundary circulation contour for that time interval, as measured by a planimeter. By making a number of test runs at different temperatures, curves can be drawn which show the percentage of circulation at any temperature for any given time interval for that particular oil.

TABLE I.—OIL CIRCULATION IN AXLE WITH ALTERNATING HIGH AND LOW PRECHILLING TEMPERATURES.

Lubricant H.

TEST TEMPERATURE, DEG. FAHR.	PRECHILLING TEMPERATURE, DEG. FAHR.	CIRCULATION IN 4 MIN., PER CENT	CIRCULATION IN 10 MIN., PER CENT
30	175	85	100
48	70	1	1
40	175	85	100
40	70	1	2
49	175	89	100
50	70	2	3

AERATION

Relatively early in the test work it was found that the oil temperature reached during the prechilling run had a profound effect upon the distribution characteristics of certain of the oils after chilling. With other oils, changes in prechilling temperatures from 50 F. to 175 F. had no effect. Figures 2 and 3 show the difference in distribution characteristics of one oil (lubricant H) at 50 F. after prechilling temperatures of 70, 100, and 125 F., respectively. The same data amplified by other tests on this same oil are given in Figs. 4 and 5; they show the profound effect of the temperature of the oil at the time axle rotation is stopped and chilling is begun. With this particular oil, having an A.S.T.M. pour point of 30 F., 67 per cent circulation was set up in 10 min. at 20 F. with a prechilling temperature of 175 F. while less than 5 per cent circulation was set up in 10 min. at 50 F. with a prechilling temperature of 70 F. The positiveness of this action is illustrated in Table I which is a record of consecutive runs on the same lubricant with alternate prechilling temperatures of 70 and 175 F.

Only data from one oil have been used in this presentation since it is manifestly impossible to present all of the many records taken; moreover, the additional data simply would be variations of the same picture.

The phenomenon is not simply a wax structure effect as was at first thought. Carrying the axle through the same temperature cycle by means of external heating and chilling without rotation of the gears did not produce the change in channeling temperatures that had been noted when the gears were in operation. Further it cannot be due to a destructive churning of the wax structures set up by the temperature changes, since such an effect would be the reverse of that observed; evidently it is an air-oil emulsion peculiar to certain types of lubricants.

The effect of prechilling temperature is a condition that any bench channel test must reproduce. Its importance can hardly be questioned, for one need only visualize the winter use to which his own family car is put, with its many short trips in which the temperature of the oil frequently will not rise above 70 or 80 F. during cold weather. It takes on added significance when it is realized that prolonged axle operation, even at rotative speeds up to 3000 r.p.m. did not provide additional distribution after the aerated condition had once been set up. Thus, unless this phenomenon is taken into consideration in establishing a safe channel temperature, one short trip may set up a condition which will result in a destructive lack of lubrication at open-road speeds, a serious condition with our present highly loaded, sensitive axle gears.

At this point a criticism of the test procedure might be raised since the test oil was originally chilled to or below its pour point even though the safe channeling temperature was found to be considerably above the pour point. If the oil had not been chilled below some intermediate point, a lower channel temperature might have resulted. To remove this uncertainty, channel tests were repeated with two of the oils showing this phenomenon, the test procedure being changed only in that the temperature of the oil was not reduced below the previously determined channel point. The distribution results were substantially identical with those obtained with the lower chilling temperature.

Obviously, in a study of this kind, it would have been unwise to depend upon the performance of the oil in one axle only. Two were, therefore, included in the original program, a spiral bevel axle and one of the smaller worm axles. It was thought that the flatter contour of the worm axle housing might present a more serious channeling than the steeper sided bevel gear housing. Surprisingly, this was not the case, due in large measure to the greater churning action of the broad worm wheel. The channeling temperatures in the worm axle were actually lower, by from 6 to 14 deg. Fahr. than in the bevel gear axle. Later a third axle, a large hypoid unit, was included in the test work, when channeling information was required on certain experimental oils before a satisfactory bench test had been developed.

CIRCULATION LIMITS

Naturally some definite degree of circulation had to be set up as a minimum. For the purpose of this work 40 per cent of the oil in movement after ten minutes of axle operation was taken as minimum satisfactory circulation, and the temperature at which this degree of circulation occurred was taken as the channel temperature of the oil. This selection seems logical, for in all three axles the oil splash to the differential carrier bearings began with about 40 per cent of the oil in movement. Further, with this rate of circulation increase, the initial supply of oil to the gear mesh was adequate and the oil reached the front pinion bearing within 2 to 4 min. A rate of circulation build up of much less than 20 per cent of the oil in motion after 10 min. is dangerous for then there is a marked tendency for the oil picked up by the ring gear to pile up upon the undisturbed oil during runs of short duration, 5 min., leaving a decided channel at the gear. When this occurs on several successive runs a complete cessation of circulation results. Actually the widest difference in temperature between 20 per cent and 40 per cent circulation in 10 min. was 6 deg. Fahr.

CHANNEL POINT BENCH TESTS

In setting up the axle channeling temperatures to be used as the basis for the development of a bench channel test, the results obtained with the worm axle were disregarded. It was felt that the more serious condition presented by the bevel gear axle should be the one considered. The channel temperatures in Table II are those obtained upon the two bevel gear axles.

A number of different types of bench tests were tried before the present method was developed. These included a so-called flat pan test, a "U" tube test utilizing the British Air Ministry cold test apparatus and the ball test as suggested by Mr. E. F. Bittner.[3] None of these tests appeared to hold much promise even when the aeration phenomenon was disregarded and provision for aeration of the oil appeared so difficult that the work with them was discontinued. However, it was noted that in general the A.S.T.M. pour test[2] gave a directional, although not an actual, agreement with the axle when aeration was not present; further the apparatus employed in this test was such as to lend itself to an aeration operation. Pour-point equipment, moreover, had the decided advantage of being universally available. The work was, therefore, directed along that line and what we feel to be a satisfactory test was finally developed.

TABLE II.—IDENTIFICATION OF TEST OILS AND A COMPARISON OF THEIR AXLE AND BENCH TEST CHANNEL TEMPERATURE.

Lubricant Designation	Gravity, deg. A.P.I.	Saybolt Universal Viscosity, sec.		A.S.T.M. Pour Point, deg. Fahr.		Axle Channel Temperature, deg. Fahr.		Bench Test Channel Temperature, deg. Fahr.	Base	Characteristic
		130 F.	210 F.	Max.	Min.	40 per cent Circulation, 10 min.	20 per cent Circulation, 10 min.			
A........	21.3	400	73.2	−5	−5	−16	−19	−20	Naphthenic	Low pour, medium low viscosity
B........	30.6	49.8		+20		+12	+11	+15	Paraffinic	High pour, low viscosity
C........	20.2	526	96.6	−5		−9	−13	−10	Naphthenic	Low pour, intermediate viscosity
D........	26.4	652	122	+5	+5	−7	−9	+5	Paraffinic	Low pour, high viscosity
E........	18.2	1389	169	+15	+15	+12	+7	+10	Blend Paraffinic and Naphthenic	High pour, high viscosity
F........	25.7	134	50	+30	+30	+19	+18	+30	Paraffinic	High pour, low viscosity
G........	22.1	905	137	+65	+35	+65	+61	+65	Mid-Continent	High pour, high viscosity
H........	25.5	755	140	+30	+25	58+	58+	+55	Paraffinic	High pour, high viscosity
I........	23.6	1483	204	+10	+10	+15	+13	+15	Mid-Continent	High pour, high viscosity
J........	24.5	2318	309	+25	+25	+38	+35	+40	Paraffinic	High pour, high viscosity
K........	22.0	1618	216	+55	+30	+63	+58	+65	Paraffinic	High pour, high viscosity
L........	27.4	386	92	+5	+5	+32	+26	+30	Blend Paraffinic and Mid-Continent	Low pour, intermediate viscosity
M........	23.4	408	88.5	−15	−15	+8	+2	+10	Blend Paraffinic and Naphthenic	Low pour, intermediate viscosity
N........	30.7	635	118	−15	−20	−29	−31	−30	Synthetic	Low pour, high viscosity

The test is simply an elaboration of the standard A.S.T.M. pour test,[2] incorporating an aeration operation to reproduce the condition observed in the axles.

The equipment for the channel point test embraces the regular A.S.T.M. pour test bath, pour point test jar and thermometer, and in addition a stirring apparatus, a special stirrer with temperature control bath and a horizontal single cell constant-temperature bath.

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[3] E. F. Bittner, "A Method of Testing Low Pressure Characteristics of Oils and Greases," *National Petroleum News*, Vol. XXIV, No. 42, p. 31.

In carrying out the test the oil is charged to the test jars following the A.S.T.M. pour-point procedure, with the exception that the special stirrer is placed in the jar instead of the thermometer. The oil is then preheated to 115 F. and chilled to its pour point as prescribed by the A.S.T.M. pour-test procedure. Since the pour point of the oil will not be known, as a rule, and to provide the standard cooling rate, a companion sample in a second test jar is simultaneously chilled for a standard A.S.T.M. pour-point determination. The latter sample acts as a monitor for changing the channel-point sample from one cooling bath to another as the oil temperature is lowered.

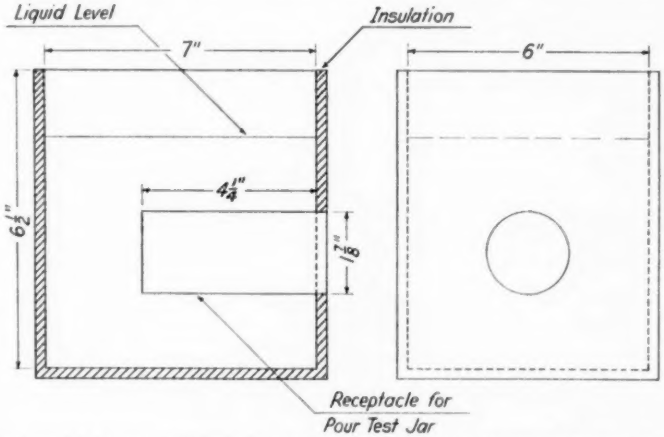

FIG. 6.—Horizontal Constant-Temperature Bath to Maintain Constant Sample Temperature During 1-min. Observation Period.

When the channel-point sample has reached the pour-point temperature as indicated by the monitor sample, the test jar is removed from the cooling bath and placed in the stirring apparatus, where air is whipped into the oil for a period of 20 min. During the stirring operation the water bath surrounding the oil sample bottle is maintained at 55 F. to prevent the oil temperature from exceeding 70 F. The temperature of 70 F. was selected since it was indicated by the axle work as the critical prechilling temperature.

At the end of the stirring period, the special stirrer is replaced by the standard A.S.T.M. pour-test thermometer and the sample jar placed in the A.S.T.M. pour-test bath at −30 F. From this point the test again follows the A.S.T.M. pour-point test procedure until the solid point is reached, with the exception that the final interval for no movement is increased to 1 min. If no movement of the oil in the sample jar is observed during this 1-min. period, the channel temperature is taken at 5 deg. Fahr. above the temperature of the oil.

Due to the fact that the oil temperature will rise when holding the jar in a horizontal position at room temperature for a period of much over 10 sec., a special low-temperature horizontal bath is used to maintain the oil at the test temperature during the 1-min. observation period. The bath contains a jacket similar in size to that specified for the A.S.T.M. low-temperature bath but is mounted in a horizontal position, Fig. 6.

The form of the stirrer is quite critical. A number of stirrers of different designs were tried before one was found which

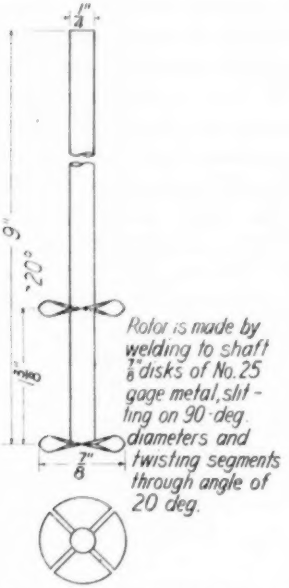
1/4"
9"
20°
1 3/8"
7/8"
Rotor is made by welding to shaft 7/8" disks of No. 25 gage metal, slitting on 90-deg. diameters and twisting segments through angle of 20 deg.

FIG. 7.—Special Stirrer Used in Aeration Operation of Channel-Point Test.

The dimensions and position of rotors are important.

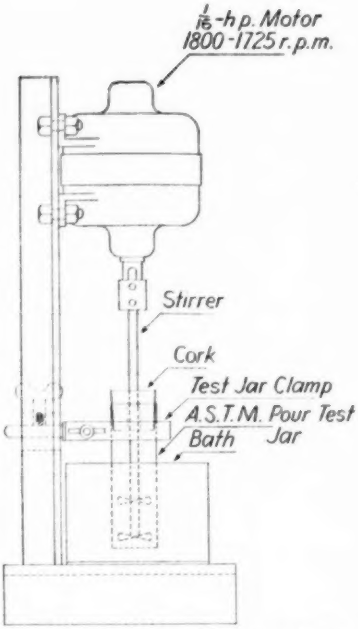

FIG. 8.—Stirring Apparatus Showing Sample in Position for Aeration Operation

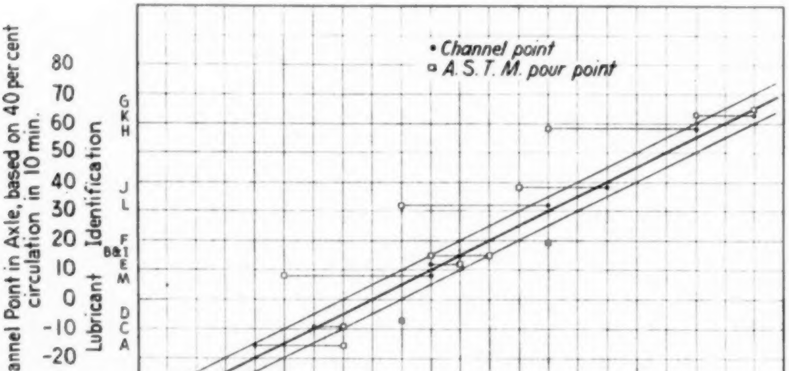

FIG. 9.—Correlation Curve Between Channel Point Temperatures by Channel Point Test and in Axle.

gave sufficient agitation to provide the same degree of aeration obtained in the axle. This is the stirrer illustrated in Fig. 7. Other than for the stirrer itself, the design of the stirring apparatus is not critical, except that the electric motor be sufficiently powerful to maintain its speed with the heavier lubricants at low temperatures. In the equipment which we employ the motor is a $\frac{1}{6}$-hp., 1800-r.p.m., single-phase unit. The motor mounting, test-jar clamp, etc., are shown in Fig. 8.

A detailed description of the apparatus and method is given in the appendix.

CORRELATION

In Table II are given the pour points and channel points of the test oils as determined by the axle tests and the channel-point test. The same information is presented in curve form in Fig. 9, the latter providing the correlation picture at a glance. The variation of the channel point test temperatures from those determined in the axle were within 5 deg. Fahr. for twelve of the fourteen oils tested. The two exceptions were 7 deg. Fahr. outside this range but the error in both cases is such that the oils would not be selected for temperatures below that at which they would function successfully. Further, one of these oils was too light to be classed as a gear oil.

We believe that this degree of correlation is commercially satisfactory; it is superior to that of the conventional pour point or to channel temperatures as determined by other methods which we have tried, and we recommend the method as a means for determining this previously all-too-elusive oil characteristic.

APPENDIX

PROPOSED METHOD OF TEST FOR CHANNEL POINT OF GEAR LUBRICANTS

1. The channel point of a gear lubricant is the lowest temperature at which the oil will flow when it is chilled without disturbance after aeration under definite prescribed conditions.

APPARATUS

2. The test jar shall conform to the requirements of A.S.T.M. Standard Method of Test for Cloud and Pour Points (D 97 - 34).[4]

3. The thermometer shall conform to the requirements of A.S.T.M. Method D 97.

4. The cork shall have a central bore $\frac{1}{4}$ in. in diameter for a distance of $\frac{3}{8}$ in. from top of cork, balance to be $\frac{3}{8}$ in. in diameter.

5. The jacket shall conform to the requirements of A.S.T.M. Method D 97.

6. A disk cork or felt $\frac{1}{4}$ in. thick and of the same diameter as the inside of the jacket will be required.

7. The ring gasket shall conform to the requirements of A.S.T.M. Method D 97.

8. The cooling bath shall conform to the requirements of A.S.T.M. Method D 97. For determination of channel points below 50 F., two or more baths should be at hand. The required bath temperatures may be maintained by refrigeration if available, otherwise by suitable freezing mixtures.

9. A horizontal bath with the same jacket as described in item 5 shall be provided to maintain oil temperature during flow observation (see Fig. 6).

10. The stirrer consists of a double propeller attached to a $\frac{1}{4}$-in. rod 9 in. long and shall conform to the dimensions given in Fig. 7.

11. A $\frac{1}{6}$-hp., 1800-r.p.m. motor with suitable stand, base, and clamp shall be

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[4] 1936 Book of A.S.T.M. Standards, Part II, p. 853.

provided to drive the stirrer, as illustrated in Fig. 8.

METHOD

12. Pour the oil into two test jars to a height of not less than 2 in. nor more than 2¼ in. One test jar shall then be tightly corked, carrying the thermometer in a vertical position in the center of the jar, with the thermometer bulb immersed so that the beginning of the capillary shall be ⅛ in. below the surface of the oil. The other test jar shall be tightly corked, this cork carrying the stirring rod immersed in the oil in a vertical position such that the bottom vanes of the stirring rod rest on the bottom of the jar. The top vanes of the stirring rod will then be approximately ½ in. below the surface of the oil.

The two samples are then heated without stirring to a temperature of 115 F. in a bath maintained at a temperature not greater than 118 F. The oil shall then be cooled to 90 F. in air or in a water bath, approximately 77 F. in temperature. Oils on which a pour point below −30 F. is expected shall be heated as above with the high-pour-test thermometer in position, cooled to 60 F. and the low-pour-test thermometer placed in position and the assembly placed in the cooling jacket.

The two test jars are then inserted in the jacket which is surrounded with a liquid at a temperature of 30 to 35 F. Not more than 1 in. of the jacket shall project out of the cooling medium.

Beginning at a temperature of 20 deg. Fahr. above the expected pour point, at each thermometer reading which is a multiple of 5 deg. Fahr., the test jar containing the thermometer shall be removed carefully from the jacket and shall be tilted just enough to ascertain whether there is a movement of the oil in the test jar. The complete operation of removal and replacement shall not require more than 3 sec. The sample jar containing the stirrer is not removed from the jacket when inspecting the oil for movement. If the oil has not ceased to flow when its temperature has reached 50 F. both test jars are placed in a second bath maintained at 0 to 5 F. If the oil has not ceased to flow when the temperature has reached 20 F. both test jars are removed to a bath at −30 to −25 F. At −10 F. should the oil still be flowing both test jars are inserted in a bath at −60 F. At no time shall the cold test jar be placed directly in the cooling medium. As soon as the oil in the one test jar does not flow when the jar is tilted, the test jar shall be held in a horizontal position for exactly 5 sec. as noted by a stop watch or other accurate timing device and observed carefully. If the oil shows any movement in the test jar under these conditions, the test jar shall be immediately replaced in the jacket and a test for flow repeated at the next temperature 5 deg. Fahr. lower.

The test shall be continued in this manner until a point is reached at which the oil in the test jar shows no movement when the test jar is held in a horizontal position for 5 sec. During the above inspections the test jar containing the stirrer has not been disturbed with the exception of transferring the jar from one bath to another. As the oil in this jar has been subjected to the same temperature conditions as that in the jar on which the pour was determined, it is reasonable to assume the oil temperatures are the same. The purpose of using two test jars is to prevent condensation from forming on the surface of the oil in the test jar, thereby giving erroneous results, should the thermometer and stopper be removed for the insertion of the stirring rod.

13. The second test jar which is now at the cold point of the oil and which contains the stirring rod is removed from the low-temperature bath and placed in the stirring bath at 55 F. The stirrer stem is inserted in the coupling attached to the stirring motor. The test jar is clamped in a position such that the bottom vanes of the stirring rod are approximately ⅛ in. above the bottom of the jar. The top vanes, being 1⅜ in. above the bottom vanes, are about ¼ in. below the surface of the oil. The motor is started and run for a period of 20 min., the stirring bath temperature being held at 55 F. As the oil becomes aerated, in light-colored oils a very definite yellow air-oil emulsion is in evidence. On the black oils the air-oil emulsion does not change the color of the oil, but very small air

bubbles cause the oil to foam. During the 20 min. of stirring the oil temperature shall not rise above 70 F. (bath temperature should be lowered if necessary). The test jar is now removed from the stirring apparatus, stirring rod removed and the pour test thermometer carrying the test-jar cork inserted in the jar.

14. The test jar is now replaced in the 30 F. cooling bath. The standard cooling procedure of Section 10 of Method D 97[4] is followed down to the cold point of the aerated oil, except that the final flow observation shall be made over a period of 1 min. During this observation the test jar is placed in the horizontal bath, which should be held at cold-point temperature, to prevent warming of the oil by atmospheric heat. Inspection for flow will be made at 30 sec. and exactly 60 sec. Should movement of the oil be noted at either the 30-sec. or 1-min. point, the test jar shall be immediately replaced in its correct low-temperature bath and chilled another 5 deg. Fahr. This procedure shall be followed until no motion is noted during the 1-min. period the test jar is in the horizontal bath. The reading of the test thermometer at this temperature, corrected for error if necessary, shall be recorded. The channel point shall be taken as the temperature 5 deg. Fahr. above this solid point.

DISCUSSION

MR. FRANK GOTTSCH.[1]—I should like to ask Mr. Stewart whether this method would be of any use in examining greases for gear cases. We have had a lot of trouble in the winter with all the apparatus that is used to clean the streets. This has to be maintained in spite of cold weather, and sometimes the gear boxes are not tight, especially the transmission gear boxes, and we must use a grease, rather than an oil as recommended. It is very important that channeling should not take place. We were forced to devise a method of our own so that this could be measured. The method was a modification of the A.S.T.M. pour point method on a large scale, that is, without aeration, and we succeeded in finding a certain size of brass tube that would fit inside of another certain size of copper pipe so that one could be slipped off the other. We filled up the inner tube with the grease and chilled it in an apparatus equipped with a cooling bath. We had a large vessel wherein we could lift up the pipe and measure the amount of flow of the grease down from the top by filling up the space thus formed with pre-cooled kerosine. We call this a "cold flow" method. It was devised to imitate the A.S.T.M. pour point method; also to make some measure of the amount of flow. This was done by holding the cooling bath between zero and 5 F., and having a thermometer centrally located in the midst of the grease 2 in. from the bottom; when the thermometer in the grease reached 20 F. we lifted the tube up and observed the amount of flow in 3 min. Whether this would be of any use to us now in view of your observations about aeration is the question. We should like to know whether we would have to aerate the grease before we made this large-size pour-point test on a transmission grease.

MR. J. P. STEWART.[2]—I should like to be able to answer Mr. Gottsch's question specifically but I cannot. We have not tried this test with grease.

In an axle even at normal temperatures (70 to 80 F.) it takes a long time for most greases to reach 40 per cent or even 20 per cent circulation. One must expect rather limited circulation with a grease. However, this limit circulation will persist through quite a temperature range, in which respect grease differs from a fluid lubricant. For this reason we do not believe the channel test as described in the paper can be applied directly to greases.

Whether aeration affects the distribution of a grease we are not in a position to say since greases were not included in the test work. It would seem that a process analogous to the aeration phase of the test should be included in a grease test, however, to give the product the worked consistency which it will reach in the axle and which will modify its distribution characteristic over that of an unworked sample.

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THE DESIGN OF ASPHALT MIXTURES FOR UNDERWATER CONSTRUCTION

By Rossiter M. McCrone,[1] and F. C. Field[2]

Synopsis

The paper sets forth the progress made in developing asphalt mixtures for mass construction above and below the water line with especial reference to jetty work in the United States. The work so far performed has demonstrated it to be practicable technically and economically to design mixtures of asphalt and local materials which, when properly placed and compacted, will congeal into a dense homogeneous concreted mass of adequate stability and toughness, highly resistant to water, and hence to attack by wave action.

Underwater construction presupposes conditions completely dissimilar to those encountered in asphalt pavement work and the paper attempts to show the purpose of the structures for which asphalt mixtures for underwater placement have been devised. Composition of mixtures materially different from those used for pavements is indicated. They should be mixed and placed at relatively high temperatures (450 F.) but the completed structure probably will meet a narrow range of temperature, probably 32 to 80 F. Impact as conceived in pavement design is not a factor. Mixing, handling, placing, and compacting asphalt mixtures in great masses under conditions met with in underwater construction involve new methods and equipment not yet fully developed.

Introduction

Nature has provided numerous natural harbors with stable entrance channels which require no artificial protection such as those which serve Boston, New York, Philadelphia, Baltimore, San Francisco, and Seattle. There are, however, many terminal ports throughout the world where artificial harbors have been created by breakwater construction, or where natural, or dredged, entrance channels are protected by jetties. Important artificial harbors include those at Los Angeles, Valparaiso, Pernambuco, Dover, Marseilles, Algiers, Naples, Bari, Alexandria, and Port Said. American harbor entrance channels protected by jetties include, among others, Ocean City, Md.; St. Johns River, Miami and St. Andrews Bay, Fla.; the Mississippi River Passes; Sabine Pass, Galveston Harbor, Aransas Pass and Brazos Island Pass, Tex.; Humbolt Bay, Calif., and Columbia River and Umpqua River, Ore.

Generally speaking, breakwaters or jetties in shallow depths, less than 40 ft., are constructed as rubble mounds with or without fascine or rubble mattresses to stabilize the foundation. The accepted practice has been to sink a foundation mattress and place thereon a core of rocks weighing 15 lb. to 2 tons and encase this core with cap and slope

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stones weighing more than 6 tons. For Gulf Coast jetties it has been customary to require that cap and cover stones be placed and bedded individually to produce a section 10 to 15 ft. wide at elevation + 6 ft. with side slopes of two vertical on three horizontal. Jetties on the Atlantic and Pacific coasts of the United States have been built similarly but on these works the cap and slope stones generally are placed on the shoulders to seat themselves by the combined forces of gravity and wave action.

Jetties restricting the width of an entrance channel should facilitate maintenance of channel dimensions by tidal flow, protect the channel from degradation by cross channel wave action, and prevent passage of sand carried alongshore by littoral currents from entering the jettied channel. The advantages of the loose rubble mound section are that it may be adapted to any site regardless of foundation conditions as settlement or loss of any section does not affect the rest of the structure and repairs are comparatively easy to make. The disadvantages are that it only partially confines the tidal flow, permits the passage of sand through the jetty into the jettied channel, and requires continuing ordinary maintenance because of degradation of section due to ordinary seas and expensive reconstruction after severe storms.

Obviously, it is desirable that jetties should be solid structures to accomplish their purpose. It is also desirable that where such structures are sited on sand beaches they should be built of the beach sand as the nearest and cheapest material. Recent developments in the use of hot asphaltic mixtures in large mass indicate the feasibility of combining beach sand with asphalt cement and a suitable mineral filler to produce a jetty structure substantially monolithic.

Materials Available.—On the Gulf Coast, rock suitable for jetty construction is distant. Generally, however, the jetties are sited on beaches which afford a bountiful supply of beach sand satisfactory for asphalt mixtures. The surveys made to locate a suitable filler for asphalt mixtures for the Mississippi River asphalt revetment work disclosed the fact that loess, occurring in the bluffs along the river from Cairo, Ill., to the Louisiana state line, is a superior mineral filler for asphalt mixtures which may be moved to all Gulf Coast jetty sites by water. Asphalt refineries are located on tide water at Texas and Louisiana points, making it possible also to deliver asphalt cement to jetty sites by water transport.

Initial Experiment.—The first opportunity for exploration of the feasibility of using asphaltic mixtures in large masses above and below water for construction or repair of marine structures such as jetties became available in connection with capping a portion of the South jetty at Galveston, described by Wilby.[3] Preliminary to this work, analyses were made of the aggregate and filler materials available for producing asphalt mixtures as follows:

ANALYSIS—GALVESTON BEACH SAND—SPECIFIC GRAVITY, 2.634

Retained on sieve No. 28, per cent...	0.05
Retained on sieve No. 35, per cent...	0.25
Retained on sieve No. 48, per cent...	0.10
Retained on sieve No. 80, per cent...	7.10
Retained on sieve No. 100, per cent...	39.7
Retained on sieve No. 200, per cent...	52.3
	0.5
	100.0

LOESS—MISSISSIPPI RIVER AND BLUFFS—SPECIFIC GRAVITY, 2.62

Retained on sieve No. 200, per cent...	0.25
Retained on sieve No. 325, per cent...	1.50
Passing sieve No. 325, per cent.......	98.25
	100.00

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[3] F. B. Wilby, "Rubble Jetties Made Solid with Asphaltic Concrete," *Engineering News-Record*, August 20, 1936, p. 263.

The properties of the asphalts used are set forth in Table I.

Design of Mixtures.—Repairs to the Galveston South Jetty were required because the structure had been flattened by wave action so that capstones of the jetty were separated by wide voids rather than by interstices. Obviously, it was desirable to design a mixture, the "seal mix," to flow through and fill the voids above and below the water line and to bind the jetty stones into a compact base and a second mixture "cap mix" to be placed on the base and build up the structure to the specified grade. After extensive laboratory research had been made, a mix was selected as the most promising for penetration through voids between cap stones into and through interstices between the smaller core stones at and below the water line and a second mix was selected as the most promising for building up the jetty structure and resisting attack by wind and weather even when exposed to summer sun and winter chill. These mixes, proportioned by weight, were as follows:

SEAL MIX

	PER CENT
Galveston beach sand	58
Loess filler	20
Asphalt cement 60 to 70 penetration	22

To be placed at a temperature of 450 F. ±25 deg. Fahr.

CAP MIX

	PER CENT
Galveston beach sand	67
Loess filler	20
Asphalt cement 30 to 40 penetration	13

To be placed at a temperature of 450 F. ±25 deg. Fahr.

Physical Properties of the Mixes.—Samples of the mixes were tested at the laboratory of the Asphalt Institute by the Hubbard-Field methods[4, 5] with results as follows:

SEAL MIX—BRIQUETS RECOMPRESSED AT 200 F.
Tamping only 60/20.

	Specific Gravity	Stability at 140 F., lb.	Specific Gravity	Stability at 100 F., lb.	Specific Gravity	Stability at 80 F., lb.
	1.930	100	1.887	550	1.893	1100
	1.894	150	1.924	750	1.895	1250
	1.906	100	1.931	650	1.898	1200
Avg.	1.907	116	1.914	650	1.895	1183

NOTE.—Pointer did not fall off when maximum stability was reached.

CAP MIX—BRIQUETS RECOMPRESSED AT 300 F.
Tamping only 60/20.

	Specific Gravity	Stability at 140 F., lb.	Specific Gravity	Stability at 100 F., lb.	Specific Gravity	Stability at 80 F., lb.
	1.970	4200	1.949	6600	1.967	over 10,000
	1.991	4600	1.951	7100	1.984	
	1.967	4000	1.954	7200	1.961	
Avg.	1.976	4267	1.951	6970	1.971	

UNDERWATER PLACEMENT

As noted in Wilby's article,[3] hot-mix asphaltic concrete was placed in deep water to restore breached sections at the outer end of the jetty. A modified mixture for this purpose was selected with a view to developing a mixture which would fall into the sea as a cohesive mass more plastic than the cap mix used inshore and of stiffer consistency than the seal mix. The mix selected, proportioned by weight, was as follows:

DEEP WATER MIX

	PER CENT
Galveston beach sand	69.0
Loess filler	13.5
Asphalt cement 30 to 40 penetration	17.5

To be placed at temperature of 450 F. ±25 deg. Fahr.

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[4] Prévost Hubbard and F. C. Field, "A Practical Method of Determining the Relative Stability of Fine-Aggregate Asphalt Paving Mixtures," *Proceedings*, Am. Soc. Testing Mats., Vol. 25, Part II, p. 335 (1925).

[5] Prévost Hubbard and F. C. Field, "A Study of Certain Factors Affecting the Stability of Asphalt Paving Mixtures," *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part II, p. 577 (1926).

Close to 200 tons were placed in one mass in a break in the jetty where the depth of water ranged from 8 to 16 ft. This mass was placed in about 2 hr. continuous operation with a $\frac{3}{4}$-yd. clamshell bucket. Tests on this work are scheduled to be made in the early future, probably during the summer of 1937.

Columbia River South Jetty.[6]—In August, 1936, a consolidation of the outer end of this rubble mound structure was undertaken.

This is a variable of the Galveston offshore work. In either case the principal involved is the same in that heat is depended upon to cause plastic flow and adequate bond. For this reason, the weight of unit charge of hot mix must be proportioned to the heat loss encountered during flow.

Table I.—Asphalt, Properties as Specified for Galveston Work.

Homogeneity determinations were made as outlined by G. L. Oliensis, "A Qualitative Test for Determining the Degree of Heterogeneity of Asphalts," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 715 (1933).

	30 to 40 Penetration Asphalt		60 to 70 Penetration Asphalt		Methods of Sampling and Testing
	Min.	Max.	Min.	Max.	
Softening Point deg. Fahr.	132		130		A.S.T.M. Method D 36 - 26
Specific Gravity	1.025		1.01		A.S.T.M. Method D 70 - 27
Ductility at 32 F.[a] at 0.25 cm. per min.	3.5		6.5		A.S.T.M. Method D 113 - 32 T
Ductility at 77 F. at 5.0 cm. per min.	100		100		A.S.T.M. Method D 113 - 32 T
Penetration at 32 F., 200 g., 60 sec.	10		15		A.S.T.M. Method D 5 - 25
Penetration at 77 F., 100 g., 5 sec.	30	40		70	A.S.T.M. Method D 5 - 25
Penetration at 115 F., 50 g., 5 sec.		180		250	A.S.T.M. Method D 5 - 25
Flash (Cleveland open cup) deg. Fahr.	500		480		A.S.T.M. Method D 92 - 24
Paraffin holde, per cent		2.5		2.3	Kansas City Testing Lab. Method No. 29[b]
Loss on Heating, 50 g. 5 hr., 325 F., per cent.		0.15		0.5	A.S.T.M. Method D 6 - 30
Penetration of residue at 77 F., per cent	80				A.S.T.M. Method D 5 - 25
Viscosity at 275 F., sec.	400		200		A.S.T.M. Method D 88 - 33
Solubility					A.S.T.M. Method D 165 - 27

[a] Ductility at 32 F. shall be 10 per cent of Penetration at 77 F.
[b] Kansas City Testing Laboratory, *Bulletin No. 25*, p. 694 (1926).

The skip charge of hot mix used was 10 tons. The depth to water, varying with the tide, indicated a dry flow of 17 to 26 ft. The 10-ton charge was broken up into numerous channels by the rock structure causing lateral flow that greatly increased the travel to water level. The desired penetration to 10 ft. below low tide could not be attained with this charge. It is felt that larger unit charges and more rapid placement would increase the penetration below water and better bond would be developed.

Experimental Construction of an All-Asphaltic Concrete Jetty Section.—As an extension of the experimental capping of the Galveston South Jetty with asphaltic concrete, an experiment was carried out in July, 1936, to show whether or not a jetty may be constructed by the use of flexible asphalt mats for toe protection, precast regular triangular prisms of asphaltic concrete for side slopes and hot asphalt mix for the main body of the jetty. It was specified that this section should be built about 30 ft. into the Gulf from the South Jetty at a point where the least depth of water during construction of the experiment was 5 ft. The asphalt mixing plant used for the capping work was made available, together with a steel oil barge, a small crawler crane, and trucks, tugs and locomotive crane as required for short periods.

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[6] R. E. Hickson, "Columbia River South Jetty Terminal," *The Military Engineer*, Vol. XXIX, No. 164, p. 135 (1936).

Reinforced flexible asphalt mats 2 in. by 18 by 18 ft., of the standard type used for mattresses on the Mississippi River,[7] were cast in a 6 high stack on a wooden floor laid on the oil barge, kraft paper being used to separate the mats. Regular triangular prisms of asphaltic concrete 3 ft. high and 6 ft. long, were cast in forms near the asphalt plant. When cooled, after 60 hr., the prisms were removed from the forms and stored in the yard until required at the jetty, to which they were moved by truck. Mats and blocks were cast of hot mix of the "cap mix" type used on the jetty capping work.

Mats were cut to 9 by 18 ft. at the jetty, lifted from the barge and swung across South Jetty by the crawler crane and laid on the Gulf bed to extend outward from the toes of the experimental section. Triangular prisms then were placed by the crane at the inner edge of the mats to box in the space to be filled by the body of the jetty section to the height of the prisms, 3 ft. The box so formed was then filled with the hot mix dumped from overturning buckets. Prisms then were placed on the filled box to enclose a second box or lift ex-

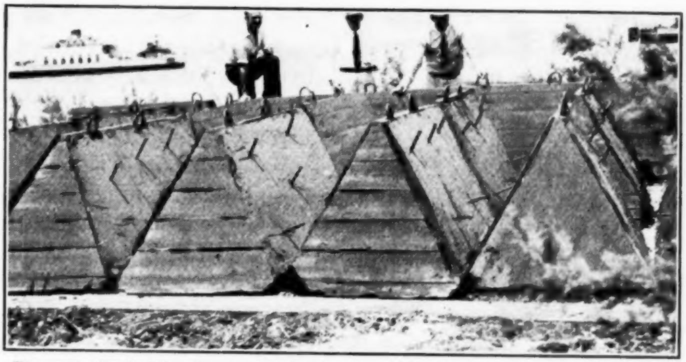

FIG. 1.—Precast Asphaltic Concrete Blocks for Side Slopes in Underwater Construction. 3 ft. high by 6 ft. long, weight 1.9 tons Galveston Jetty.

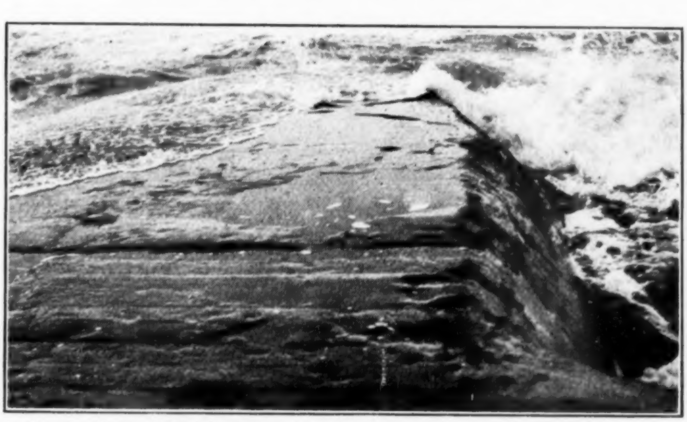

FIG. 2.—Surface of Finished Structure Galveston Jetty Before Filling Cracks.

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[7] Walter C. Carey, "Development of the Asphalt Mattress," *The Military Engineer*, Vol. XXVII, No. 156, November–December, 1935, p. 430.

tending a foot above water and this box was filled similarly with hot mix. Mixes used on these underwater lifts were of the underwater type developed on the Galveston Jetty work. Dependence was placed on the weight of superposed mass for compaction. A third line of blocks was then placed to carry the side slopes up to elevation 5 ft. sloped as necessary to meet the jetty crown and the space enclosed was filled with hot mix of the cap-course type. Vibrators were used for compaction and the surface was finished with the vibrating screed. Shortly after completion of the experiment, cracks developed across the body of the section. Surveys disclosed that the outer edges of the flexible mats had turned down and were bedded in the sand floor and that the triangular prisms were in place. The cracks obviously were caused by contraction of the body of the structure as the lower uncompacted portion was consolidated by the superposed mass. The cracks were filled and did not reform. It is expected that cores will be taken to determine the internal structure of this experimental section. It was in good condition at the latest report.

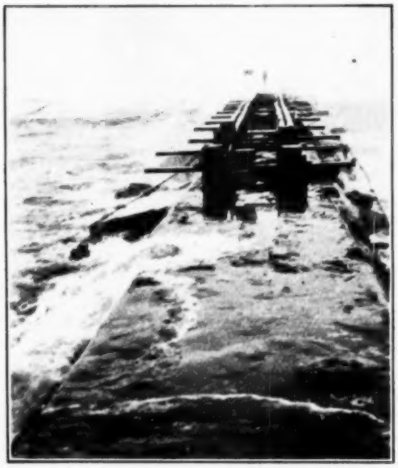

FIG. 3.—Finished Jetty Section—Runway Construction for Extension.

Placement.—Proper proportioning of suitable materials is a prime requisite in designing asphalt mixtures for mass construction above and below water, but a satisfactory design must provide also for developing proper methods of placement and for adequate compaction to build up a structure highly resistant to attack by natural forces of waves and weather. Such a structure should be a dense, homogeneous, tough mass with adequate stability but without excess rigidity. Top and side slopes must be especially well compacted to form waterproof surfaces at temperatures ranging from 32 to 140 F.

It is not possible to define an ideal mix or ideal materials. The properties of mixtures suitable for mass construction above and below water may be described only as disclosed by the experimental work so far performed. There is a reasonable range for proportioning materials for mixtures suitable for mass construction above and below water, but always the mixtures must be handled, placed and compacted at temperatures in excess of 400 F.

The Galveston work indicates that the use of hot asphalt mixtures for underwater construction involves displacement of water by a series of cohesive masses designed to be placed so that they retain sufficient internal heat to consolidate adjoining masses and form a homogeneous solid even under water.

In capping the Galveston South Jetty, work was started inshore and the asphalt mixture, dumped on the structure, was distributed and the side slopes built up by hand shoveling. The material was compacted, not very satisfactorily, by steam driven internal vibrators and the top and side slopes were finished by vibrating screeds and irons heated by the exhaust steam. After passing the shore line it soon became necessary to do all work practically from the runway over the jetty, and building and compacting the side slopes became increasingly difficult and the resulting surface became more and more unsatisfactory. It became evident that for general use of hot asphalt mixtures under water, a different method should be devised for construction of satisfactory side slopes. The use of precast units of asphaltic concrete, preferably triangular prisms, which may be inspected before placement to make certain that satisfactory side slopes are exposed is believed to be the key to successful use of hot asphalt mixtures for underwater construction. It may be noted that when such prisms are placed to enclose a space which then is filled with hot asphalt mix in units large enough to convey sufficient heat, the masses will consolidate with each other and with the confining lines of prisms even under water.

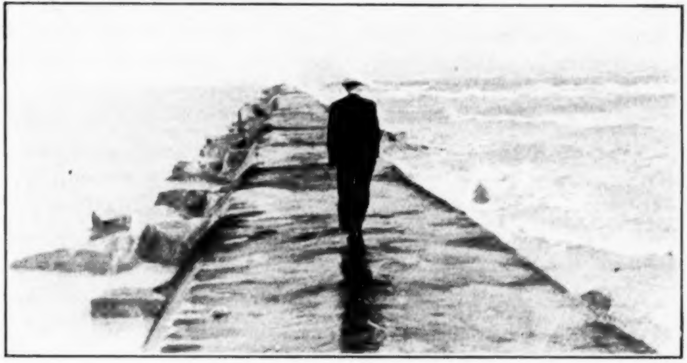

FIG. 4.—Completed Jetty.

Compaction.—The experimental work at Galveston disclosed that the lower portions of deep masses are compacted by the superposed mass. The upper portion may be compacted by vibration but this essential feature of this type of construction has not been solved completely. The difficulties of operating vibrating tools in hot asphalt mixtures at temperatures ranging from 300 to 450 F. are obvious. There are some promising lines of tools but the market has been so small that there is little incentive for manufacturers to construct tools for such exacting service.

DISCUSSION

The several projects undertaken have indicated what may be expected as to the behavior of hot asphalt mixtures used for mass construction above or below the water line and has disclosed certain principles of design of mixtures and precautions as to placement and compaction which may be summarized as follows:

Asphalt mixtures suitable for this exacting service must be designed so as: (*a*) to withstand mixing at 450 F., (*b*) to withstand, in large mass (100 tons minimum), storage in insulated barges for a minimum of 72 hr. (*c*) to be handled direct from plant by truck, skip or car, and from storage barge by clam shell into skip. Such a mixture, when dumped from skips, shall fall free as a cohesive mass to remain intact while falling through air and water to its place of deposit, (*d*) to retain sufficient internal heat to weld adjoining units of mass, and (*e*) to congeal as a dense mass when compacted by superposed loading or by vibration, or by both.

The development of this use of asphalt mixtures so far has not disclosed a method for securing compacted side slopes for material deposited under water. These slopes, however, may be formed satisfactorily by the use of precast units of asphaltic concrete which may be cast, compacted, and inspected on shore and placed to retain and consolidate into the body of the structure. Preferably, these units should be regular triangular prisms which are essentially stable and which, when consolidated into the structure, produce an economical section.

Asphalt mixtures, used for underwater construction, should have adequate stability and maximum toughness rather than rigidity, within the range of water temperature to which they may be exposed. Asphalt mixtures used for flexible mats for toe protection or shore revetment, for precast prisms for side slopes, and for the cap portion of the structure should produce a surface of maximum density that is highly water resistant.

The work so far performed has demonstrated the practicability of designing suitable mixtures of asphalt with materials available locally for mass construction above and below the water line. Further development may be expected to crystallize methods of placement and compaction and extend the field of construction with asphalt mixtures in unconfined seawater.

DISCUSSION

Mr. S. L. Damon[1] (*by letter*).—As to jetty construction on the North Pacific Coast, it is no longer the practice to use a mattress to stabilize the foundation, as additional stone necessary to compensate for possible loss of bottom material can probably be placed at less cost than the mattress. Also in large jetties which are common to this region the base of the structure may be 200 ft. or more in width, and the placing of mattresses to this great width in rough water would involve construction difficulties. It may also be stated that sand movement through these large jetties has been found to be negligible. They are from 100 to 150 ft. in width at the water line and more at the base and with small stone and marine growth in the interior are, for practical purposes, sufficiently sand tight. Sand undoubtedly passes through or over the rubble jetties of small section, and there appears to be a good field for the use of asphaltic mixtures in tightening such structures, and to bind the stones together.

The use of asphalt at the outer end of the large Columbia River jetty to prevent movement of the enrockment by the heavy winter seas has so far not proved to be successful. This jetty is 26 ft. high above low water, and it appears that the asphaltic material in passing through the attenuated passages between the large stones, with cold wet surfaces even above low-water level was chilled and transformed into a brittle porous mass with little adhesive or binding qualities. The subsequent action of the winter seas working on the lower levels picked out or moved the unbound stone, leaving a vertical face, and the bound material in the upper portion broke down.

It is not believed, as suggested in the paper, that pouring the asphaltic mix in larger volume per charge would have materially changed the results in this case. While the skip charges were of 10 tons each, three were dumped in rapid succession, making 30 tons poured at a time at any point. A large part of the pour remained on top of the enrockment for several minutes while it was worked into the voids, and it does not appear that any practical increase in the volume dumped at any time would materially affect the conditions in the lower levels. A total of 12,000 tons of mix was placed in the outer 300 ft. at the end of the jetty in the period August 5 to September 11, 1936.

Deep penetration, to the extent used on this job does not appear to give satisfactory results, but it may be possible if the stone work were built up in layers, each impregnated before the next was placed, that sufficient bond would be secured. This however appears doubtful in the rough cold water of the North Pacific Coast.

Messrs. Rossiter M. McCrone[2] and F. C. Field[3] (*authors' closure, by letter*).—The work done to date indi-

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[3] Chemist, The Asphalt Inst., New York City.

cates, it is believed, that the successful use of hot asphalt mix for mass construction above, and especially below the water line, particularly with respect to existing jetties and similar marine structures built of rubble stone on sand foundation, depends on proper proportioning, heating and mixing the materials, placement of the hot mix at high temperatures as nearly as possible in its final position, and adequate compaction by vibration or by pressure of superposed masses. The considered opinion of the authors is that the stability of such a structure necessarily involves stabilization of the foundation at and outward from the toes of slopes, construction thereon of side slopes adequate to withstand attack by wave and weather, and proper placement and compaction of hot mix between the side slopes and the core structure. It is believed that stability may not be expected to result from simple impregnation or grouting of an enrockment. This process was tried at Reservation Point, Los Angeles, without success before it was attempted on the Columbia River jetty to which Captain Damon refers.

With reference to the desirability of mattress protection under and outward from the toes of such structures, it is noted that such protection was favored by the Sixteenth International Navigation Congress at Brussels in 1935. (Reference may be made to Conclusion IX (c) for the Second Question, Second Section, Ocean Navigation, of that Congress.)

In locations such as the North Pacific Coast of the United States where rock may be placed at low cost, it may be possible economically to produce jetties capable of withstanding the heavy seas by placing additional rock to permit the formation of a stable structure by action of natural forces.

The use of asphalt mix for construction of jetties presupposes conditions where such structures are required at locations where the foundation is sand, and rock is not available at low cost. Under this condition it is beleived that all possibilities should be exhausted to develop methods for construction of durable structures by the use of the sand on which, or in which, the structures must rest.

COMPRESSION TESTING OF ASPHALT PAVING MIXTURES—II

BY ROLAND VOKAC[1]

SYNOPSIS

A brief review of flow measurement indicates that asphalt mixtures are most likely quasi-viscous or plastic systems. Consideration of the fundamental relationship of shear stress to rate of shear, by means of which flow properties of materials are evaluated, leads to an analogous relationship of compressive stress to unit rate of volume displacement measured in the compression test. Data are offered which appear to satisfy the conditions of the assumed analogy. Methods by which flow properties can be measured in a compression test using either variable rates of speed in testing or various dimensions of specimen are suggested.

Last year a paper was presented before the Society regarding our earlier experimental work on compression testing of asphalt paving mixtures at room temperatures.[2] The data offered show that the compression test measures physical characteristics such as compressive strength, elastic limit, modulus of elasticity, and modulus of permanent deformation (that is, resistance to flow after compressive strength has been exceeded). These characteristics are generally understood by the engineering profession and the use of a compression testing machine is familiar to anyone interested in strength of materials.

STRESS IN ASPHALT SURFACES

Following the preliminary work considerable attention was given to developing a correlation between data obtained in the laboratory by means of the compression test and the behavior of the mixtures tested as observed in surfaces under actual field conditions. In the course of this study several other testing methods were also investigated. The findings are available elsewhere[3] and therefore a detailed discussion will not be offered here. The correlation of the data indicates that the compression test was found more sensitive to the factors affecting service behavior than the other tests used.

We believe this to be particularly significant in view of the observations made by others with respect to resistances developed in flexible surfaces. Housel[4] when discussing the structural design of flexible pavements has said, "From a review of the existing information on pressure distribution, it is concluded that unless a minimum thickness of mat equal

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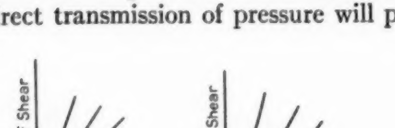
[1] Technical Bureau, Research Division, The Barber Co. Inc., Maurer, N. J.

[2] Roland Vokac, "Compression Testing of Asphalt Paving Mixtures," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 552 (1936).

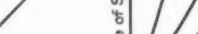
[3] R. Vokac, "Correlation of Physical Characteristics with the Service Behavior of Asphaltic Mixtures," *Proceedings*, Technical Sessions, Assn. Asphalt Paving Technologists, New Orleans, La., January 1937.

[4] W. S. Housel, "Design of Flexible Surfaces," *Proceedings*, Twenty-third Annual Highway Conference, University of Michigan, Ann Arbor, Mich., February, 17, 1937.

to one-quarter of the width of the contact area is used, the full surface pressure is transmitted to the sub-grade." Taking Gray's[5] figures, the equivalent diameter of the average contact area of tires with wheel loads varying from 2000 to 10,000 lb. will vary between 5.6 and 12.0 in. Paraphrasing Housel's observations to suit these figures it is apparent that with small tires, surface pressure will be transmitted directly to the subgrade or base when the surface is not more than $\frac{5.6}{4} = 1.4$ in. in thickness. With the larger tires the same kind of direct transmission of pressure will prevail in surfaces up to $\frac{12.0}{4} = 3.0$ in. in thickness. It is safe to say that with average tires and loads, surfaces up to $2\frac{1}{4}$ in. in thickness allow direct transmission of surface pressure to the base. It is not surprising, therefore, that in our studies we have found compressive resistance, which represents the conditions of direct transmission of pressure, to give good indications of service performance in sheet asphalt paving mixtures which are seldom laid to depths greater than $1\frac{1}{2}$ or 2 in.



(a) VISCOUS FLOW (b) QUASI-VISCOUS FLOW



(c) PLASTIC FLOW

FIG. 1.—Types of Flow.

FLOW IN ASPHALT MIXTURES

There is also another phase to the problem of the service-strength relationship of asphalt paving mixtures which should be developed. Those who have observed the performance of asphalt pavements over periods of years can recall instances of mixtures which exhibit adequate strength by any test, but which have gradually developed rutting for no apparent reason. More frequently we have all seen the condition where an asphaltic surface is carrying the heaviest kind of rapid-moving through traffic without sign of displacement, but which bears the imprint of the tires of any vehicle which has stood parked for a comparatively short time. The answer to the problems suggested by these conditions is unquestionably associated with time, or in other words, duration of application of stress.

Thus, a complete understanding of the service performance of an asphalt paving mixture from a purely physical aspect must eventually resolve itself into a study of flow properties of the paving mixture.

In the present paper we wish to discuss the use of the compression test for the study of flow properties of asphalt paving mixtures. Observation of asphalt mixtures shows that most of them are quasi-viscous or plastic materials, and the extent to which they are stressed determines the rate of flow. The three recognized types of flow are "distinguished by the nature of the curve obtained by a plot of rate of shear *versus* shearing stress"[6] (see Fig. 1). This assumes that the rate of shear is

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[5] B. E. Gray, "Principles of Design of Flexible Type Pavements," *Proceedings*, Technical Sessions, Assn. Asphalt Paving Technologists, January 1937.

[6] R. N. Traxler, "Influence of the Solid on the Flow Properties of Dilute Suspensions," *Paper Trade Journal*, Vol. 104, pp. 151–154 (1937); *Chemical and Metallurgical Engineering*, Vol. 44, pp. 200–203 (1937).

determinate, that is, the direction and magnitude of shear can be evaluated in every element of a test specimen. Descriptions of the various types of instruments employed for the measurement of flow may be found in the literature.[7,8] All are so constructed that simple laminar flow exists throughout the specimen.

Viscous flow is represented when a plot of rate of shear *versus* shearing stress gives a straight line passing through the origin as in Fig. 1 (*a*). The reciprocal slope of the straight line, which is constant over the entire range of shearing stress, evaluates the viscosity of the material, that is, viscosity = shearing stress/rate of shear. In the testing of viscous materials the rate of shear must always be kept low enough to avoid turbulence.

The type of curves obtained with systems possessing so-called quasi-viscous flow characteristics are illustrated in Fig. 1 (*b*). Plots of rate of shear *versus* shearing stress for these materials give various curved lines passing through the origin; the "viscosity" calculated decreases with increasing shear stress. When a "viscosity" value is reported for such a material, the shearing stress or rate of shear used must be reported also. Quasi-viscous substances contain a sufficient concentration of dispersed particles to impart structure to the system. Such systems are frequently encountered in asphalt-filler mortars, blown asphalts, and the like. It is also probable that many asphalt mixtures will be found of this type.

The general relation that exists between rate of shear and shearing stress in plastic flow is the equation of a straight line, as in Fig. 1(*c*), which intersects the shearing stress axis at some point called the yield value. Stress below the yield value will not deform the material permanently. The slope of the straight line after the yield value is exceeded is known as the mobility of the system. However, this only represents the ideal case; at low rates of shear the curves may bend sharply and pass through the origin as indicated by the dotted lines in Fig. 1(*c*) instead of intersecting the shearing stress axis. A yield value is generally recorded, however, since this point and the slope of the line define the behavior of a plastic material.

Determination of rates of shear and shearing stress in cylindrical specimens under compressive loads resolves itself into a complicated problem as investigators have found.[9] The greatest obstacle is in determining satisfactorily the position of the planes upon which the shear takes place during flow under axial compression. The theoretical values, derived from a consideration of laminar flow, are approximated by the data only when very thin samples are tested. It has also been found extremely difficult to correlate the results with those obtained in other instruments used for the measurement of flow. Therefore, it appears that if the compression test is to be used at all in the study of flow of asphalt mixtures, some other basis for evaluating flow properties must be used.

Fundamentally, the rate of shear in any present-day method of viscosity measurements is always a proportional function of the rate of volume displacement of the sample in the test. Thus, in the capillary tube method[10] the volume of material moved is measured at definite

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[7] R. N. J. Saal, "Determinations Regarding the Plastic Properties of Asphaltic Bitumen," *Proceedings*, World Petroleum Congress, Vol. 2, p. 515 (1933). Published in London, England.

[8] R. N. Traxler and H. E. Schweyer, "Measurement of High Viscosity—a Rapid Method," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 518 (1936).

[9] R. L. Peek, Jr., "Parallel Plate Plastometry," *Journal of Rheology*, Vol. 3, pp. 345–372 (1932).

[10] E. C. Bingham, "Fluidity and Plasticity," McGraw-Hill Publishing Co., New York City (1922).

time intervals and from this the rate of shear can be evaluated; in the falling coaxial cylinder[5] the movement of the inner cylinder is a proportional function of the volume of material displaced, and so on. Since this relationship appears to be fundamental to measurement of flow we have considered the following analogy.

In the compression test we can easily measure the volume of material displaced by measuring the travel of the moving head with respect to the fixed head during the test of a specimen. From this, the unit rate of volume displacement may be determined, which is analogous and most probably a direct function of the rates of shear existing in the specimen during the test:

$$R = \frac{vA}{h} \ldots\ldots\ldots\ldots\ldots\ldots (1)$$

where R = unit rate of volume displacement in cubic inches per inch per minute,

v = velocity of one head of the testing machine with repect to other in inches per minute,

A = original area of cylindrical cross-section of briquet in square inches, and

h = original height of specimen in inches.

Continuing this reasoning further, we recognize the fact that compressive stress may always be resolved into two components originating from shear resistance within a material. It should follow therefore that compressive stress, p, is some direct function of the shear stress in an asphalt mixture. An equation describing a plastic substance may be written:

$$p = a\,\frac{vA}{h} + K$$

or,

$$p = a\,R + K \ldots\ldots\ldots\ldots (2)$$

where p = compressive stress in pounds per square inch,

a = a constant representing "mobility," and

K = factor representing the "yield strength."

It will be shown later that the factor K is influenced by the dimensions of the specimens tested. For the present, however, it will be considered as a constant.

DATA AND DISCUSSION

By way of testing the basic assumptions in the foregoing development, mixtures were made up into briquets 2 in. in diameter by 1 in. tall and tested at several constant rates of deformation. Typical data are shown in Table I. Separate briquets were tested at each of the rates indicated and each resulting ultimate compressive stress was taken as the value for p. This is contrary to the usual procedure in viscometry where a constant stress is applied to the material and the rate of shear is measured. This procedure is followed only for convenience, however, and the reverse order of manipulation is equally possible.

TABLE I.—TYPICAL TEST DATA.

Constant D/h = 2.00.

Mix A[a]		Mix B[b]	
$\frac{vA}{h} = R$	p	$\frac{vA}{h} = R$	p
0.019	261	0.031	560
0.025	316	0.079	648
0.031	328	0.13	674
0.045	401	0.17	709
0.062	488	0.22	828
0.094	546	0.27	828
0.12	621	0.36	865
0.25	826	0.41	941
0.36	1000		

[a] Cold mix, freshly made, contains 1 per cent naphtha. Trinidad asphalt cement, component, 60 penetration.

[b] Hot sheet asphalt mixture. Trinidad asphalt cement, component, 60 penetration.

The data in Table I have been plotted in Fig. 2. If our analogy is sound the curve for mix A represents a quasi-viscous material and the curve for mix B a plastic material. This seems to be a true definition of each. Mix A is a freshly made cold mixture containing about 1 per cent of solvent (naphtha) which was not given time to evaporate. Mix B is a hot sheet asphalt mixture of conventional design. Both mixes contain 10 per cent of bitumen from Trinidad asphalt cement of 60 penetration and 10 per cent of limestone dust filler. The sand is bank sand from a local source.

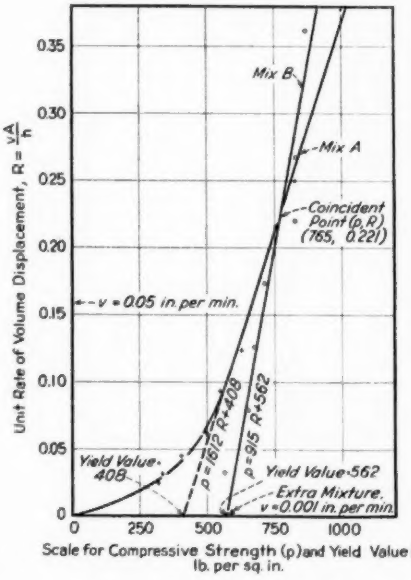

FIG. 2.—Data from Table I.

The figure is of particular interest in the fact that at a rate of volume displacement, R, of 0.221, the curves intersect showing a compressive stress of 765 lb. per sq. in. Among other things this fact should serve to indicate the fallacy of ordinary testing methods, wherein only a single rate of test is used to determine a factor of strength in an asphalt mixture. For instance, at a value $R = 0.157$, resulting from a testing speed v of 0.05 in. per min. on briquets 2 in. in diameter by 1 in. tall, the values of p are nearly alike whereas the full range of each curve indicates mix A will flow even when stressed very lightly, while mix B would offer considerable resistance to the same flow.

In order to check the apparent continuation of the straight line toward the stress axis, an extra point was determined on a mixture of the same composition and ingredients as used in mix B. This mixture was tested at the slowest rate possible with our machine. The rate of deformation was $v = 0.001$ in. per min. The maximum compressive stress was recorded in about 90 min. as $p = 585$ lb. per sq. in., $R = 0.003$ cu. in. per inch per min. Plotting this point in Fig. 2 finds it in substantial agreement with the rest of the data from mix B. It may be said, therefore, that mix B represents within all practical limits the ideal case of a plastic material, and we should expect this mixture to withstand 560 lb. per sq. in. compressive stress with no appreciable flow.

TABLE II.—DATA ON SERIES OF BRIQUETS WITH D/h APPROXIMATELY 2.00.

Rate of volume displacement varied over same range for three diameters of briquets.

Briquet[a]	h	D/h	$v \times 10^{-2}$	R	p
No. 1	0.54	2.09	15.1	0.28	1190
No. 2	0.56	2.02	12.6	0.23	990
No. 3	0.53	2.12	7.56	0.14	754
No. 4	0.53	2.12	5.04	0.09	878
No. 5	0.56	2.02	2.52	0.05	668
No. 6	0.52	2.17	1.26	0.02	587
No. 7	0.78	2.04	10.1	0.26	940
No. 8	0.78	2.04	8.82	0.22	930
No. 9	0.79	2.01	6.30	0.16	828
No. 10	0.79	2.01	5.04	0.13	852
No. 11	0.80	1.99	2.52	0.06	691
No. 12	0.78	2.04	1.26	0.03	630
No. 13	1.13	2.00	7.56	0.27	984
No. 14	1.13	2.00	6.81	0.24	882
No. 15	1.14	1.98	6.05	0.21	870
No. 16	1.13	2.00	3.02	0.11	841
No. 17	1.14	1.98	1.51	0.05	745
No. 18	1.13	2.00	0.76	0.03	689

[a] Diameter of specimens Nos. 1 to 6 = 1.13 in. Area = 1.00 sq. in.
Diameter of specimens Nos. 7 to 12 = 1.596 in. Area = 2.00 sq. in.
Diameter of specimens Nos. 13 to 18 = 2.26 in. Area = 4.00 sq. in.

As mentioned with reference to K in Eq. 2, further experiment has shown that the intercept is influenced by the dimensions of the test specimen. Tables II and III contain data illustrating this effect. The data in both tables have been obtained on briquet specimens of the same mixture. This was a hot sheet asphalt mixture prepared in our $\frac{1}{2}$-cu. ft. laboratory pug mill mixer. Its composition was as follows:

Sand, per cent	80
Limestone dust, per cent	10
Oil asphalt cement (60 penetration), per cent	10
	100

TABLE III.—DATA ON SERIES OF BRIQUETS MADE WITH SAME MIXTURE AS THOSE IN TABLE II.

v only held constant. $\frac{D}{h}$ and $\frac{vA}{h}$ vary according to dimension.

Briquet	h	D/h	p	$v \times 10^{-2}$	R	$1090R$	K
Nos. 1 to 6[a]	0.57	2.00	858	10.08	0.18	196	662
No. 19	0.83	1.36	664	10.08	0.12	131	533
No. 20	1.12	1.01	484	10.08	0.090	98	386
No. 21	1.39	0.81	402	10.08	0.073	80	322
No. 22	1.69	0.67	352	10.08	0.060	65	287
No. 23	2.24	0.51	311	10.08	0.045	49	262
Nos. 7 to 12[b]	0.80	2.00	868	7.56	0.19	207	662
No. 24	1.19	1.34	686	7.56	0.13	142	544
No. 25	1.58	1.00	506	7.56	0.096	105	401
No. 26	1.99	0.80	454	7.56	0.076	83	371
No. 27	2.40	0.66	369	7.56	0.063	69	300
No. 28	3.25	0.49	258	7.56	0.047	51	207
Nos. 13 to 18[c]	1.13	2.00	836	4.54	0.16	174	662
No. 29	1.69	1.34	654	4.54	0.11	120	534
No. 30	2.28	0.99	463	4.54	0.080	87	376
No. 31	2.88	0.79	414	4.54	0.063	69	345
No. 32	3.42	0.66	382	4.54	0.053	58	324
No. 33	4.04	0.56	329	4.54	0.045	49	280

[a] Diameter of specimens in this group = 1.13 in. Area = 1 sq. in.
[b] Diameter of specimens in this group = 1.596 in. Area = 2 sq. in.
[c] Diameter of specimens in this group = 2.26 in. Area = 4 sq. in.

The data in Table II are for three sets of mixes made into briquets with diameters 1.13, 1.596, and 2.26 in., respectively. (These diameters give cross-sections of 1, 2 and 4 sq. in., respectively.) The diameter to height ratio is constant within practical limits for the three sets. The velocity of deformation, v, is varied so that the rate of volume displacement, R, covers approximately the same range in each set. Grouping the data from the first three briquets of each set and the last three of each set, a straight line was calculated for the data by the method of averages using Eq. 2 for the relationship of p and R. (Data on briquet No. 1 was omitted since comparison with No. 7 and No. 13 shows this value of p to be too great.) Constants a and K were evaluated with the following result:

$$p = 1090\,R + 662 \quad \text{(3)}$$

The average deviation of the data is found to be ±5.67 per cent.

The data for p and R in Table II and the straight line represented in Eq. 3 are plotted in Fig. 3 with reference to the scale on the right-hand side of the graph.

In Table III are data obtained on the same mixture when D/h and R are both variable over approximately the same ranges for each diameter of specimen.

Using the value of $a = 1090$ for the "mobility" factor as determined in Eq. 3, the value of K has been calculated for the data on each mix in Table III. The values obtained for K are listed in the last column, which is, algebraically:

$$K = p - 1090\,R \quad \text{(4)}$$

It will be noted that these calculated values of K run in descending order in each set in a manner proportional to D/h. Plotting the values of D/h against K in Fig. 3 indicates a straight-line relationship which can be expressed as follows:

$$K = b\frac{D}{h} + c \ldots\ldots\ldots\ldots (5)$$

Evaluating the constants b and c in this equation with the data in Table III results in

$$K = 292\frac{D}{h} + 106 \ldots\ldots\ldots (6)$$

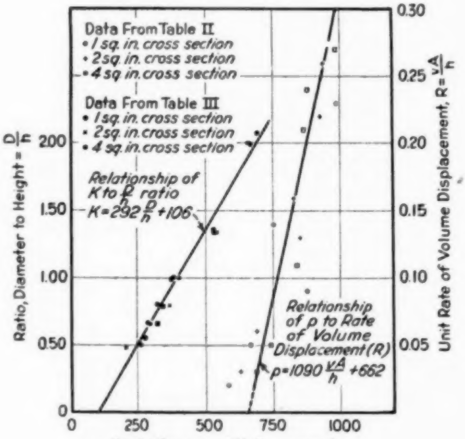

FIG. 3.—Data from Tables II and III.

The average deviation of this data is ±4.85 per cent.

Equation 5 may be substituted in Eq. 2 to give the general equation for plastic flow measured by the compression test as follows:

$$p = aR + b\frac{D}{h} + c \ldots\ldots\ldots (7)$$

where p, R, D and h are the usual variables, and a, b, c are constants depending on the flow properties of the material.

The values of constants a, b and c obtained by Eqs. 3 and 6 may be substituted in Eq. 7 to evaluate the present mixture as follows:

$$p = 1090\,R + 292\frac{D}{h} + 106 \ldots (8)$$

Constant a in Eq. 7 is a function of the "mobility" of the mixture as in Eq. 2. The entire expression ($b\,D/h + c$) now represents the "yield value" K. It is evident that when D/h is held constant this expression becomes a constant and the relationship expressed in Eq. 2 may be used for evaluating flow.

Of particular significance in analyzing Eq. 7 is the fact that variation of the dimensions of the specimen changes the position of the intercept but does not affect the slope. This is equivalent to saying that although varying the dimensions of a specimen will change its "yield value," such variation will have no effect whatsoever on the "mobility" of the mix. Evidently the dimensional relationship D/h largely determines the ability of a mixture to withstand loading without deformation.

CONCLUSION

The foregoing analysis indicates a wider field of application for the compression testing of asphaltic mixtures. The analogy of compressive stress to shearing stress, and rate of volume displacement to rate of shear, is apparently sound as evidenced by the type of curves obtained with the compression data. For engineering purposes it is more convenient to deal in terms of the applied compressive load and volume displacements than to use the more scientific consideration of rate of shear and shear stresses. The present method of test suggests the possibility of relating flow properties of the finished mixture with those of its fluid component.

From the practical standpoint of testing, a standard D/h value should be adopted for testing all mixtures. If this is done, Eq. 2 may be used to evaluate the data. This would require determination of two, or preferably more, points using different values of R in order to determine the slope a and intercept K. Of particular advantage too is the fact that R may be varied in either of two ways: (1) the rate of deformation v of the sample may be varied, or (2) the specimens may be made of various dimensions (so long as the D/h ratio is kept constant) and the several samples may be tested at one rate of deformation. In the first case the velocity v would be the variable of the test; and, in the second case, both diameter D and height h would be varied in like proportions. All of this is readily adaptable to routine testing as well as to research. A machine capable of operating at only one speed suitable to the test may be used if one large and one small diameter briquet are made. Where variable speeds are available, single size specimens will suffice.

DISCUSSION

Mr. W. S. Housel.[1]—In the recent development of testing methods employed in measuring the physical properties of semi-cohesive materials such as soils or bituminous mixtures, there are two problems which appear to be of primary importance. In very general terms these problems are: first, to devise test methods which properly evaluate the time element; and second, to devise ways and means of determining the effect of dimensions of the test specimen in relation to the actual physical property being measured. The author has made a very substantial contribution in connection with both of these problems.

The necessity for ingenuity in the isolation of different variables, such as employed by the author, is nowhere more evident than in the various shear tests that are in the process of development at the present time. Some shear tests employ a constant rate of loading, some a constant rate of deformation, and some apply load by progressive increments with sufficient time for each load increment to come to a point of equilibrium or a uniform rate of deformation. One sees reference in current literature to a rapid shear test and a slow shear test with a variation in results on the same materials which makes definite conclusions a hazardous estimate.

Presumably the engineer interested in predicting the behavior of a road surface, a subgrade, or a foundation is most often interested in static resistance and must determine that resistance which is available to produce a condition of permanent equilibrium independent of the time element. The author has arranged the variable factors resulting, after analysis, in a simple linear relation between the time element and compressive strength, which makes possible a determination of the actual yield value in a positive manner. The necessity of conducting tests at several rates of shear in order to evaluate the time relation is clearly shown and, although it should be obvious, seems to require some emphasis. A similar method of attack appears to have value in measuring any type of resistance, although the factors involved may not be the same in other cases.

The author adopts very much the same line of reasoning in evaluating the effect of dimensions on the compressive strength of the mixture. His factor K in Table III and Fig. 3 corresponds to the yield value or ultimate shearing resistance in a cohesive material or the ultimate stability of a granular material. The data indicate that the compressive strength decreases directly with a decreasing ratio of diameter to height of cylinder to a constant value at which the effect of dimensions is eliminated. This result is verified by the writer's research in internal stability of granular materials in which it was found that there was a decrease in stability as the height of cylinder was increased until direct transfer of load from top to bottom of the cylinder was eliminated. The results referred to have led to an investigation of the effect of the dimensions of the specimen, which is still in progress. In

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[1] Research Consultant, Michigan State Highway Dept.; Associate Professor of Civil Engineering, University of Michigan, Ann Arbor, Mich.

general the data from it indicate a hyperbolic relation between internal stability and the ratio of height divided by diameter of the cylinder. This relation holds up to a certain h/D ratio where the stability curve becomes asymptotic to a line which represents the actual stability independent of dimensions. If the same data were plotted against the inverted D/h ratio, the linear relation shown by the author would result.

In the writer's opinion the compression test as employed by the author measures a very important property of granular mixtures and may be accurately described as the ultimate stability at zero lateral pressure. In order completely to describe granular mixtures it appears that stabilometer tests which measure change in stability at various amounts of lateral pressure are also necessary. Such tests are needed to measure the angle of pressure transmission which, in the writer's opinion, is the basic factor in internal stability.[2] After sufficient data are accumulated it may be shown that stability at zero lateral pressure or compressive strength is directly related to the angle of pressure transmission. The compressive strength test may then emerge as the final routine test, since it is much better suited for practical testing than the more cumbersome stabilometer test. This possibility is enhanced by the fact that other data presented by the author indicate a favorable correlation between compression tests and service behavior.[3]

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[2] W. S. Housel, "Internal Stability of Granular Materials," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 426 (1936).

[3] Roland Vokac, "A Correlation of Physical Tests with the Service Behavior of Asphaltic Mixtures," *Proceedings*, Assn. Asphalt Paving Technologists, p. 202 (1937).

THE HOMOGENEITY OF WEST TEXAS ASPHALTS

BY SIDNEY BORN[1]

The use of solvents to precipitate or separate certain constituents present in crude oils and bituminous substances has been known for many years. G. L. Oliensis,[2] has proposed a test for determining qualitatively the degree of "physical heterogeneity" in asphalt. The test depends upon the precipitation that takes place when such asphalts are dissolved or dispersed by 5.1 times their volume of certain petroleum naphthas. The term "carbenoids" is proposed as the name of the precipitated bodies. Oliensis proposes to use as a standard solvent for this test a material having the following specifications:[3]

> "The naphtha solvent used shall be a straight-run overhead distillate free from cracked products of any kind, and shall conform to the following requirements:
>
> Gravity, deg. A.P.I.....49 to 50
> Distillation:
> Initial boiling point..Above 300 F.
> 50 per cent over......335 to 355 F.
> End point...........Below 410 F.
> Aniline number.........138 to 145 F."

When asphalts are tested by this method they produce either a uniform brown circular stain or a brown stain with a much darker spot or nucleus in the center. Oliensis classifies asphalts which produce a uniform spot by this test as homogeneous and those with a darker spot or nucleus as heterogeneous. He concludes that this test will classify the following types of bitumens as homogeneous:

1. Steam-refined residuals known to have been refined without serious cracking.
2. The bitumen of certain native asphalts.
3. Some types of slightly oxidized residuals from asphaltic-base crude oils.

Types of bitumens classified as heterogeneous are:

4. Steam-refined residuals that have been overheated during the refining operation.
5. Cracking-coil residuals.
6. Highly blown residuals.

Oliensis has definitely pointed out that while this test should not be considered as a quality test, "it will identify all petroleum asphalts that have been subjected to temperatures higher than are normal in the regular steam-refining processes. Hence it will detect (*a*) cracked asphalts, (*b*) steam-refined or vacuum process residuals that have been accidentally subjected to higher temperatures than are normal, and (*c*) the more highly blown asphalts that have been subjected to the higher temperatures for prolonged periods of time." Later he modified these views to add that the following factors might also classify an asphalt as heterogeneous even though high temperatures or cracking were not involved:

1. Waxy bodies,
2. Acid sludge bodies,
3. The combination of incompatible

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[1] Professor of Petroleum Research, University of Tulsa, Tulsa, Okla.

[2] G. L. Oliensis, "A Qualitative Test for Determining the Degree of Heterogeneity of Asphalts," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 715 (1933).

[3] G. L. Oliensis, "A Further Study of the Heterogeneity of Asphalt—a Quantitative Method," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 494 (1936).

fractions in so-called "synthetic asphalts," and

4. Exposure to the air.

In testing certain asphalts produced from West Texas crude oils, the author noticed that some of these gave a "positive" spot test by this method. At first we attributed these spots to overheating during the process of manufacture, but a careful check of processing operations failed to substantiate this idea. In order to determine whether the positive spot test of these asphalts was caused by overheating or whether it was due to some other factor, a number of West Texas crude oils were reduced to asphalts in vacuum at very low temperatures.

These crude oils were obtained directly from the producing wells, care being taken to obtain representative samples from each of the fields selected. All of the oil-producing formations in this area are generally considered as of Permian age.

The Chalk field is located in Howard County and produces from two formations, a shallow horizon producing from 1600 to 1800 ft. and a deeper horizon producing from a depth of approximately 3000 ft. The crude oils from these two sands have quite different characteristics; the one produced from the shallow sand has a gravity of 31 to 32 deg. A.P.I. while the deeper sand produces oil with a gravity of 26 to 27 deg. A.P.I.

The Iatan field is located in Mitchell County and produces from several different strata, all within 200 ft. of each other at a depth of 2800 ft. The gravity of this oil varies from 27.5 to 29.7 deg. A.P.I.

The Westbrook field is located in Mitchell County and produces from the 3000 ft. horizon. It has a gravity of 24 to 26 deg. A.P.I.

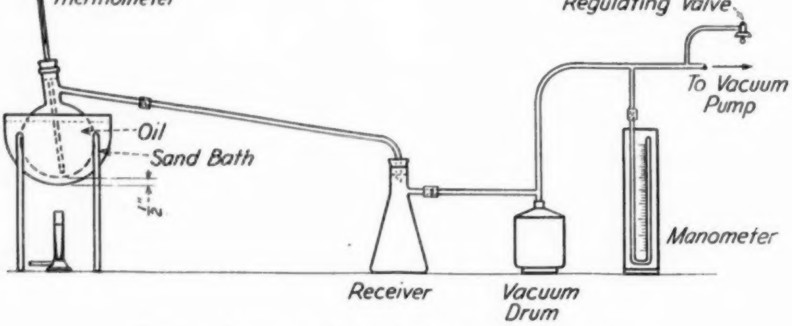

FIG. 1.—Apparatus for Reducing Crude Oil in Vacuum.

The Ector field is in Ector County, Texas, and produces oil from the 3678-ft. horizon. It has a gravity ranging from 30.8 to 33.2 deg. A.P.I.

The method used for reducing these crude oils to asphalts in vacuum was as follows: (See Fig. 1.)

Apparatus:

A.S.T.M. low-distillation thermometer.
Sand bath in metal pan.
Cutback asphalt flask.
Brass tubular air cooled condenser.
Vacuum pump and manometer.
Graduated cylinder.
Vacuum flask, 250 ml., with side arm.

Setup:

The flask-to-condenser joint, and the thermometer cork were both covered with a mixture of powdered asbestos and glue. The thermometer was inserted through the neck of the flask until the bulb projected to within approximately ½ in. from the bottom of the flask. The lower three-quarters of the flask itself was covered with fine sand to a depth of at least ½ in. The end of the condenser entered the vacuum flask through a rubber stopper, and the side arm was connected to a vacuum pump.

TABLE I.—TESTS ON NAPHTHAS PRODUCED FROM WEST TEXAS CRUDE OILS.

Distillation	Shallow Chalk 46.3 deg. A.P.I.	Iatan Crude 46.1 deg. A.P.I.	Westbrook 45.5 deg. A.P.I.	Ector 47.0 deg. A.P.I.	Deep Chalk 46.1 deg. A.P.I.
Initial boiling point......	310 F.	297 F.	300 F	307 F.	300 F.
5 per cent....	320 F.	304 F.	305 F.	315 F.	309 F.
10 per cent...	323 F.	305 F.	307 F.	317 F.	311 F.
20 per cent...	327 F.	307 F.	310 F.	320 F.	314 F.
30 per cent...	329 F.	310 F.	313 F.	323 F.	317 F.
40 per cent...	332 F.	313 F.	315 F.	327 F.	320 F.
50 per cent...	337 F.	316 F.	319 F.	331 F.	324 F.
60 per cent...	343 F.	320 F.	323 F.	336 F.	328 F.
70 per cent...	349 F.	325 F.	327 F.	343 F.	333 F.
80 per cent...	357 F.	328 F.	333 F.	351 F.	340 F.
90 per cent...	369 F.	345 F.	342 F.	365 F.	353 F.
Maximum....	407 F.	387 F.	371 F.	407 F.	391 F.
Recovery, per cent.......	98.6	99.0	99.0	99.0	99.0
Residue, per cent.......	1.4	1.0	1.0	1.0	1.0
Loss, per cent.	0	0	0	0	0
Aniline number........	124 F.	104 F.	93 F.	119 F.	108.5 F.

Procedure:

200 ml. of the crude oil to be reduced was charged to the flask, the joints glued and the sand covering the flask heaped up on the sides. Flame was applied so that the initial boiling point occurred within about 20 min., approximate A.S.T.M. distillation rate being maintained until a liquid temperature of 500 F. was reached. The receiver was emptied of low boiling materials, and vacuum was carefully applied until a minimum of 5 mm. mercury was obtained. Care was taken to prevent the liquid temperature from going over 500 F. The tendency of the whole body of residue to foam out of the flask was overcome by regulating the application of vacuum, sudden differences being eliminated by the use of a large airtight container in the vacuum line to absorb slight pressure changes.

From the initial application of the flame to the completion of the test required about 1 hr. The product obtained ranged in melting point (ring-and-ball method) from 88 to 104 F., and contained all high-boiling materials present in the crude itself (boiling over 500 F. at 5 mm. mercury pressure). The asphalt contained no "synthetic asphaltic materials" or acid sludges, and was not subjected to prolonged air or light exposure.

Some of the asphalts produced in this manner showed positive spot tests with standard Oliensis solvent. Evidently there was some other cause for this than overheating. After this had been established, we ran a number of tests to ascertain the character of the solvent present in each crude oil tested, and the influence of this solvent on the asphalt.

In the preparation of these solvents, the following equipment, setup, and procedure were used:

Apparatus:

3000-ml. ring neck round bottom flask.

Hempel type fractionating column, 1 by 15 in.

A.S.T.M. condenser, standard.

A.S.T.M. low-distillation thermometer.

Setup:

The flask, Hempel column, and condenser were assembled with corks, and 2000 ml. of the crude to be reduced was charged to the flask, the corks being glued to prevent loss of naphtha and to reduce fire hazard, and the thermometer adjusted in the neck of the Hempel column so that the top of the bulb was level with the bottom of the side arm of the column, and the bottom of the bulb just above the beads in the column.

Procedure:

A small flame was applied, so that distillation started and a rate of approximately 5 ml. per min. was maintained. Distillation was allowed to continue until a vapor temperature of 300 F. was reached, this temperature being maintained until distillation essentially stopped. The flame was cut down slightly, and the material allowed to reflux for 10 min. In a clean receiver, the naphtha distilling above 300 and below 400 F. was collected for further test. Table I gives the properties of the naphthas obtained from five different West Texas crude oils.

In addition to these five solvents, a Stoddard naphtha-toluol mixture having an aniline number of 60 F. and a similar mixture having an aniline number of 20 F. were prepared. The former solution required 45 per cent and the latter 59 per cent by volume of toluol in Stoddard solvent. Freshly distilled aniline was used in each set of determinations.

TABLE II.—RELATIONSHIP BETWEEN ASPHALT, SOLVENT, AND SPOT TEST.

	Melting Point, deg. Fahr.	West-brook Solvent	Deep Chalk Solvent	Iatan Solvent	Ector Solvent	Shallow Chalk Solvent	Stand-ard Solvent	60 F. Aniline	20 F. Aniline
Westbrook asphalt	97	N	N-P	N	N-P	P	P	N	N
Deep chalk asphalt	92	N	N	N	N	N	N-P	N	N
Iatan asphalt	97	N	N-P	N	N-P	N	N-P	N	N
Shallow chalk asphalt	88	N	N	N	N	N	N	N	N
Ector asphalt	96	N	N	N	N	N	N	N	N
Pressure tar No. 1204	..	P	P	P	P	P	P	P	P
Pressure tar No. 1205	104	P	P	P	P	P	P	P	P

P = Spot.
N = No spot.
N-P = No spot after 30 min.; spot after 24 hr.

Oliensis tests were then run on each of the five asphalts and on two pressure tars which definitely were known to have been cracked, using each of the seven special solvents, and standard solvent. The results of these tests are tabulated in Table II.

Some investigators[3] have assumed that the positive "spot" test obtained from certain asphalts which had not been subjected to cracking or high temperatures might be due to the presence of waxy bodies. Our tests failed to show any relation between the wax content of the asphalts tested and the spot test:

CRUDE SOURCE	PENETRATION AT 77 F.	WAX, PER CENT	SPOT TEST
Deep Chalk	94	2.4	Positive
Shallow Chalk	102	2.1	Negative
Iatan[a]	110	3.6	Positive
Westbrook	90	2.3	Positive

[a] Some of the crude oils from this field produce asphalts with a negative spot test.

The wax determinations were made by the usual alcohol-ether precipitation and represent an average of five tests each.

Further tests were carried out to determine the effect of adding paraffin wax to an asphalt which gave a negative spot test:

ASPHALT, PER CENT	PARAFFIN ADDED, PER CENT	OLIENSIS SPOT TEST	PARAFFIN BY TEST, PER CENT
100	..	Neg.	2.4
99	1	Neg.	3.0
98	2	Neg.	3.8
97	3	Neg.	5.0
96	4	Neg.	...
95	5	Neg.	...
90	10	Neg.	...
88	12	Neg.	...
85	15	Pos.	14.5

Obviously, these four asphalts should fall under the classification of homogeneous materials, yet three give positive spot tests with regular Oliensis solvent. They have not been cracked, and have not been air blown. As evidenced by the addition of pure paraffin wax to the asphalts and subsequent Oliensis tests, the waxy materials present in the asphalts are not present in sufficient quantities to give a positive spot test. No acid sludges were present, and exposure to the air was minimized.

This leads us to the conclusion that asphalts produced from these crude oils cannot be judged by the standard Oliensis test, with regard to cracking. Carbenoids, or those materials responsible for the formation of the spot in the test, must have been present in sufficient quantity in the original crude oil to cause a positive spot test when concentrated in the yield of asphalt. The concentration of these materials is probably due to the high solvent power of the original solvent present in the crude oil.

Consideration of the data presented here indicates that the Oliensis test as it is now used establishes a purely arbitrary line between homogeneous and heterogeneous asphalts. This method does not distinguish between asphalts which have been cracked or subjected to excessive temperatures during process of manufacture and those which due to some unusual characteristics of the crude oil itself produce a positive "spot" test. The data submitted show that such natural asphalts can be differentiated from cracked asphalts by lowering the aniline number of the solvent used so that it is equivalent to the aniline number of the natural solvent occurring in the crude oil from which the asphalt was produced.

Once the characteristics of the natural solvent from any asphaltic crude oil have been ascertained, a synthetic solvent having the same aniline number and boiling range can be used by mixing a suitable amount of xylene or toluene with the standard Stoddard solvent. These modified solvents will still detect the presence of cracked materials even when they are present in relatively small amounts.

Effect of Adding Cracked Materials on Spot Test

The base material used for these tests was an asphalt made from shallow Chalk crude oil, having a penetration of 135 at 77 F. which showed a negative spot test with standard Oliensis naphtha. The cracked asphalt added was made from West Texas crude oil.

Percentage of Cracked Asphalt No. 1 Added...	4	6	8
Naphtha Used			
114 F. aniline number.........	N	N	N
119 F. aniline number.........	N	N	N
125 F. aniline number.........	N	P	P
130 F. aniline number.........	N	P	P
Standard Oliensis solvent.....	P	P	P

In the following test the same base material was used but a cracked asphalt from Oklahoma crude oil was added.

Percentage of Cracked Asphalt No. 2 Added...	4	6	8
Naphtha Used			
114 F. aniline number.........	N	N	P
119 F. aniline number.........	N	N	P
125 F. aniline number.........	N	N	P
130 F. aniline number.........	N	P	P
Standard Oliensis solvent.....	P	P	P

These tests indicate that the use of lower aniline point naphthas will disclose the addition of comparatively small amounts of cracked asphalts.

Conclusions

Asphalts produced from pressure still tars can be definitely identified by solvents of lower aniline numbers than the standard solvent, even as low as 20 F. aniline number giving a positive test with the cracked asphalts used.

It has been definitely established that asphalts produced from certain West Texas crude oils will give a positive spot test with standard Oliensis solvent even though these materials have been produced at very low temperatures without overheating or cracking.

A method has been presented for differentiating between asphalts which have been overheated or cracked and those which give a positive spot test due to certain inherent qualities of the crude oil itself.

It has been shown that, while paraffin wax may cause a positive spot test there is no relation between the amount of paraffin in a crude oil and the results obtained by the Oliensis test. It requires the addition of paraffin in large quantities to cause a normally homogeneous asphalt to be classified as heterogeneous by this test.

DISCUSSION

Mr. G. L. Oliensis[1] (*presented in written form*).—Substantially the same data as presented by Mr. Born were known to me and were given careful study; in fact, it was precisely these data that started me on the investigation which I had reported in my 1936 paper.

The author reports that he carefully distilled in his laboratory 200 ml. of each of five West Texas crude oils, using vacuum as soon as the temperature of the liquid had been raised to 500 F., and maintained that temperature until the residue had reached a melting point of 88 to 104 F. My opinion is that (taking into consideration the extreme susceptibility to overheating that some of these West Texas crude oils display, as will be shown later) the quantity used for making this distillation, and particularly the quantity of the residue, which at the end of the test was presumably 40 ml. or so, was far too low, and the temperature of the liquid was far too high, to assure positively that no overheating or other heat breakdown could have taken place; and consequently Mr. Born cannot safely assume, as he apparently does, that no overheating did take place. Nevertheless, we note that the residues from two of the crude oils, namely Ector and shallow Chalk, proved negative to the spot test; the residues from two others, the Iatan and the deep Chalk, proved weakly positive (inasmuch as the spot was clear when fresh and became positive only after 24 hr.); while the residue from only one, the Westbrook, proved positive from the start. It is therefore quite possible that under much more careful distillation, and with the use of a much larger volume of the crude, to reduce the possibility of local overheating, the residues from the Iatan and the deep Chalk would also have tested negative at the end of 24 hr., so that only one crude oil, the Westbrook, appears definitely difficult to reduce to a negative residue.

In order to establish whether the failure of some of his five residues to yield a negative spot was connected in any way with the presence of waxy bodies, Mr. Born reports that the paraffin wax content of four of them ranged between 2.1 and 3.6 per cent. He also points out that by starting with a negative asphalt of 2.4 per cent paraffin content, and adding additional paraffin, the asphalt remained negative until between 12 to 15 per cent of the paraffin had been added, at which point it became positive. He thereupon concludes, apparently, that *any* "positive" asphalt that is found to contain less than 15 per cent paraffin by test, must be assumed to be positive through some fact *other* than waxy bodies.

A somewhat different light on this subject, however, is cast by my 1936 paper (the paragraph headed "Heterogeneity Due to Waxy Bodies"), in which is found the following statement:

> It is known . . . that these waxy bodies are better tolerated by some asphalts than by others. For example, straight paraffin wax may be safely combined in nearly all

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proportions with certain asphalts (gilsonite selects); may combine safely in certain proportions with other asphalts (Mexican or Venezuelan residuals); and may not combine at all with still other asphalts (grahamite). If combined in other than the tolerated proportions, an apparently homogeneous blend may sometimes ... be effected, which on prolonged heating in the kettle will precipitate a heavy sludge or sediment.... When asphalts contain waxy bodies for which they have a sufficiently low tolerance, they may be expected to react heterogeneous to the spot test.... When such asphalts are heated, the degree of heterogeneity may increase with remarkable rapidity until actual precipitation of relatively coarse xylene-insoluble carbonaceous bodies results.

The paper just quoted then goes on to show that such a wax-asphalt mixture could develop extreme heterogeneity when heated for as little as 20 min. at 400 F.; and would also develop extreme heterogeneity even when heated at the remarkably low temperature of 212 F.

The statement in the foregoing paper that heterogeneity is caused not by the actual percentage of solid paraffin but by the low degree of tolerance existing between any particular asphalt and its waxy bodies makes untenable Mr. Born's position when he establishes an arbitrary dividing line at 15 per cent paraffin, and declares that any positive asphalt having less than that content must be judged positive through some other cause than paraffin. Furthermore, since as little as 400 F., and even 212 F., may develop coking or some similar heat-breakdown in mixtures of asphalt and waxy bodies, once the lack of tolerance between the two has developed, I question Mr. Born's statement that no overheating or cracking whatever developed when he distilled only 200 ml. of wax-bearing crude at a temperature as high as 500 F. On the contrary, the presumption is that incipient cracking, or at any rate some type of heat-breakdown, took place in the case of at least three of the small distillations described.

Nor can I feel that the finding of 2.1 to 3.6 per cent solid paraffin in the five West Texas crude oils tells the whole story of the waxy bodies therein. As pointed out in my 1936 paper, it is recognized that the laboratory determination of the content of solid paraffin gives no indication of the more liquid paraffinaceous bodies that may also be present, and yet these latter may conceivably disturb the balance between the asphalt and the waxy components even more seriously than may the solids. It is known that many of these West Texas crude oils, notably the Westbrook, yield a heavy distillate of such high pour point that it becomes practically solid at room temperatures from the waxy bodies carried over. Therefore, such liquid waxy bodies too, even though incapable of detection by laboratory test, may contribute to the development of heterogeneity in the refining process.

Mr. Born's proposal to use naphthas of higher solvent power than the standard, in order to assure a negative spot on residues which would otherwise fail to pass, is a rather ingenious one. He would distill off the naphtha native to any given crude oil, and use only that naphtha (instead of the standard naphtha) to determine the spot test on the residues derived from that crude oil. While the specification for the standard naphtha to be used in the spot test prescribes an aniline number of 138 to 145 F., Mr. Born shows in his Table I that the naphthas derived from his five West Texas crude oils have aniline numbers ranging considerably below those figures, namely, from 124 down to 93 F. The lower the aniline number, the higher is the solvent power of the naphtha, and consequently, the greater the degree of heterogeneity (as revealed by the standard naphtha) that such a higher solvent naphtha would be able to mask or conceal. This is illustrated in Table II of Mr. Born's paper, where the standard naphtha shows that only two of the five West Texas residues are negative, whereas the two naphthas of lowest aniline number, namely, the Iatan and the Westbrook naphthas, indicate that all five of the residues are negative.

Mr. Born recognizes this situation when he states:

> Carbenoids, or those materials responsible for the formation of the spot in the test, must have been present in sufficient quantity in the original crude oil to cause a positive spot test when concentrated in the yield of asphalt. The concentration of these materials is probably due to the high solvent power of the original solvent present in the crude oil.

The following statement appears in my 1936 paper:

> Even crude petroleum underground will frequently precipitate its waxy bodies under predisposing causes, unless it contains lighter fractions of relatively high solvent power; but even in the latter case, when these lighter fractions are eventually removed in the distillation process, the residual may display its low tolerance for its waxy bodies, in the oily, waxy scum that develops on its surface on standing.

These two statements cover identically the same ground. Mr. Born's statement indicates that if the light fractions referred to had not happened to have such high solvent power, the carbenoid-like bodies would have been precipitated from the crude oil before it reached the surface. That the crude oil does actually precipitate poorly-tolerated waxy and other bodies is proved by the heavy deposits of wax, frequently found impregnating the rocky strata through which the crude oil flows, necessitating blasting operations or the use of large charges of acid to free a passage for the crude oil. If the crude oil happens to contain light fractions of unusually high solvent power, then this natural tendency for the precipitation of the less soluble bodies is prevented. Later, when these solvents are distilled off in the refining process, the low degree of tolerance between the asphalt and the waxy bodies in the solid residue is again brought into play, the mixture consequently becomes very susceptible to overheating, as brought out in the work I reported on mixtures of asphalts and waxes at 400 F. and 212 F., and eventually during the refining process, even if the latter is carried on with extreme care, a type of incipient cracking or heat breakdown does take place.

It is interesting to note, as a corroboration of the foregoing, that Westbrook crude, which yields a naphtha of the lowest aniline number (only 93 F.) and which naphtha therefore has the highest solvent power of the five, and consequently *could* have successfully combated the largest degree of incompatibility in the crude oil—precisely this Westbrook crude oil yields residues that are notoriously difficult to refine without developing a positive spot. It is the only one of the five, in fact, whose residues as prepared by Mr. Born yielded a positive spot from the start. On the other hand, at the very opposite end of the list from the Westbrook crude oil, are the Ector and the Shallow Chalk crudes, whose naphthas have the highest aniline number and therefore the poorest solvent power of the five. Hence, in the crude oil stage, natural incompatibility in these two could not have been combated so successfully and the incompatible bodies would have been rejected or precipitated. It is significant, therefore, that the residues from *these* two crude oils were found by Mr. Born himself to be absolutely negative to the standard spot test.

It seems quite clear that when crude oils contain mixtures of asphalts and waxy bodies that for some reason are incompatible, their tendency to form "positive" residues during their refining process is practically directly proportional to the high solvent power of their light fractions.

The proposal to use in the spot test a naphtha of higher solvent power than standard, in order to mask or conceal the spot, would simply defeat the purpose of the test. There is clearly no logical reason to recombine a residue with the naphtha derived from the same crude oil—just because they happen to exist in the same crude originally.

Moreover, such a proposal appears highly impractical, due to the confusion it will frequently cause. A question that will no doubt come up again and again is, for example, what type of naphtha is to be used if a residue happens to be derived from a *mixture* of two or more West Texas crude oils—particularly if the proportions of the individual crude oils in the mixture are not known, or if they may vary widely. Is it not clear that the arguments and confusion that are bound to develop on this subject will eventually utterly discredit the spot test?

That the use of these naphthas of higher solvent power would be a mistake is strikingly brought out by Mr. Born's figures (in the two tabulations under "Effect of Adding Cracked Materials on Spot Test") where the standard naphtha proves to be the only one that reveals the presence of 4, 6 and 8 per cent of cracked asphalt added to a "negative" asphalt, whereas the naphthas derived from the West Texas crude oils fail to reveal a positive spot when, in some cases, as much as 8 per cent cracked asphalt has been added. Furthermore, in those two tabulations no naphtha was used having a lower aniline number than 114 F. What is to be expected if the naphtha from Westbrook, showing 93 F. aniline number, had been used? Even higher percentages of cracked asphalt than 8 per cent could have been added with impunity.

In brief, I feel (1) that Mr. Born has not proved that the small experimental residues that he prepared in his laboratory and that tested positive had not been overheated or cracked in his distillation; (2) that whenever the straight vacuum or steam-refined residues from natural crude oils react positive when tested with the standard naphtha, these residues are heterogeneous within the meaning of the standard spot test, regardless of their showing with naphthas of higher solvent power; and moreover, a definite presumption of cracking or overheating or heat-breakdown of some type, either intentional or unintentional, also exists in such cases; (3) that to use a naphtha of higher solvent power than the standard has no basis in logic, is not consistent with our knowledge of petroleum technology and discriminates against the residues from other crude oils that do not happen to contain light fractions of equally high solvent power; and (4) that the use of naphtha of higher solvent power than the standard naphtha will impair the ability of the spot test to detect wilful cracking, or wilful adulteration with cracked material, to a much greater degree than when the standard naphtha is used.

MR. SIDNEY BORN[2] (*author's closure*).—Mr. Oliensis laid great stress upon the fact that our distillations were carried out in 200-ml. flasks: That was merely the method described in our paper as a convenient laboratory method of preparation. We have, however, substantiated these same results, not only in laboratory equipment of various sizes, but by large scale plant tests running thousands of barrels in vacuum at very low temperatures.

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[2] Professor of Petroleum Research, University of Tulsa, Tulsa, Okla.

Mr. Oliensis has repeatedly referred to the "extreme susceptibility to overheating that some of these West Texas crude oils display," but nowhere does he present any experimental evidence to this effect. Apparently he has overlooked the fact that crude oils as well as asphalts contain many different hydrocarbons and other substances, some of which are soluble in certain solvents and some of which are not. The experimental work he carried out on adding large percentages of wax to certain asphalts is not at all pertinent to this subject as the quantities of wax he used and the temperatures he employed bear no relationship to those encountered in actual practice. Furthermore in working with West Texas crude oils we have never been able to obtain "the relatively course xylene-insoluble carbonaceous bodies" that Mr. Oliensis referred to in his paper.

Mr. Oliensis, in his 1936 paper, stressed the fact that mixtures of paraffin wax and certain asphalts would develop a spot test when they were heated for periods of time, varying from 20 min. at 500 F., to 50 hr. at 210 F. In these tests he used a mixture of 25 per cent paraffin wax and 75 per cent asphalt. This percentage of wax is far in excess of any ever encountered in practice. The highest percentage of wax in any of the asphalts we tested was 3.6 per cent.

In modern asphalt practice by the use of vacuum stills, the asphalt is subjected to the temperatures we use only a fraction of a minute, in many cases only a fraction of a second. The stills are very carefully designed and the asphalt goes through at comparatively high velocities, more than 15 ft. per sec. cold in many cases. Since both time and temperature influence reactions, if one has a very short time interval and a very low temperature, one cannot have much of a reaction.

Ordinarily in petroleum work, we do not consider that any cracking takes place under 600 to 650 F. unless the oil is subjected to that temperature for very long periods of time.

Another point Mr. Oliensis raised is that our proposed method would permit the addition of cracked asphalts to straight-run asphalts or vacuum distilled asphalts. Our data show that it would in certain cases permit the addition of very small amounts of cracked asphalts, amounts that from a practical refiner's standpoint would be quite impossible even if he wanted to be unscrupulous.

I am not at all convinced that cracked asphalts are inferior to straight-run or vacuum distilled asphalts, and we may see the time when cracked asphalts will command a premium over straight run asphalts, just as cracked gasolines command a premium over straight-run gasolines.

Summing up the entire matter it is our opinion that the Oliensis test which was originally designed to differentiate cracked asphalts from steam refined or vacuum distilled asphalts tells nothing about the characteristics of asphalts except the reaction of these asphalts to a distorted test. If we would carry Mr. Oliensis' reasoning further by using solvents having a higher aniline point than those he recommends we could obtain spots with any asphalt because the entire test is based on the relative solubility of the different materials present in asphalt to a solvent which has been arbitrarily chosen.

While the present paper covers only West Texas asphalts, we now have in course of preparation another paper in which we find that this same condition that we have depicted here exists in other crude oils, notably those from Montana and New Mexico.

INDENTATION AND COMPRESSION SHEAR TESTS FOR DETERMINING SERVICE STABILITY OF ASPHALT PLANK

By H. W. Greider[1] and Henri Marc[1]

Synopsis

This paper presents a comparison of the results of two methods of indentation testing for asphalt plank: (*a*) "compression shear," run with a hydraulic compression testing machine and (*b*) loaded sphere method, using the portable indenter developed by J. W. McBurney. Data are given for eight samples of asphalt plank tested at 32, 77 and 125 F. by both methods.

Two methods of determining temperature susceptibility of plank from indentation data are presented. Limitations of field tests are brought out and determinations of hardness at a single temperature are shown to be insufficient to evaluate asphalt plank. A combination of tests is suggested to insure service stability and durability of asphalt plank used as bridge flooring.

The composition of asphalt plank determined by standard analysis is shown to be unrelated to its physical properties.

The need for high stability in asphalt plank has become apparent, since some cases of failure of this bridge flooring material by excessive rutting or by movement of the slabs under heavy or fast traffic have been noted. Various indentation tests, giving results considered as indices of "hardness," have commonly been used to determine stability and have been included in virtually all purchase specifications. These tests usually have consisted of applying a specified load to the surface of the plank through a spherical ball or a flat ended cylindrical plunger, for a stated time interval, and measuring the depth of indentation.

It had become increasingly evident that these tests did not provide enough information about the stability of asphalt plank. The authors, therefore, in 1932, developed a new method of stability testing, designated the "compression shear" test, consisting essentially of subjecting a flat-end loading tool to a load increasing at a fixed rate with respect to time. Penetration of the tool into the asphalt plank is measured for several different increments of load.

The compression shear test is most conveniently made on a hydraulic type compression testing machine. A modification of this test, adapted to the screw-type testing machine, has been adopted by the American Association of State Highway Officials for testing asphalt plank[2] at a single temperature, 125 F.

In 1934, McBurney[3] proposed an equation for relating depth of indenta-

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[1] Director of Research, and Research Chemist, respectively, The Philip Carey Manufacturing Co., Lockland, Ohio.

[2] A.A.S.H.O. Standard Specifications for Highway Materials (1935); Specification M-46, p. 61; Method T-77, p. 212.

[3] J. W. McBurney, "Indentation of Asphalt Tile," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 591 (1934).

tion to time when a constant load acts upon a hemispherically ended indenting tool. The constants in McBurney's equation are evaluated by measuring indentation at two different times. At the March 6, 1936, meeting of Subcommittee D-4 on Asphalt Planks of the Society's Committee D-4 on Road and Paving Materials at Pittsburgh, D. T. Colton of the Johns-Manville Corp. advocated the use of the McBurney indentation tester as a means of specifying stability under load. The purpose of this paper is to compare the stability results obtained on identical samples of asphalt plank by the two methods, compression shear and McBurney indentation, at 32, 77 and 125 F., and to devise measures of the effect of temperature on asphalt plank.

TABLE I.—COMPOSITION, IMPACT AND BRITTLENESS TESTS.

	Sample A-1	Sample A-2	Sample B-1	Sample B-2	Sample C-1	Sample C-2	Sample D-1	Sample D-2
Thickness, in.	1.0	1.5	1.0	1.5	1.0	1.5	1.0	1.5
Asphalt, per cent	43.6	33.0	35.7	34.5	42.9	42.7	32.3	41.7
Organic fiber, per cent	15.0	16.8	11.4	14.5	15.0	15.1	18.1	12.2
Inorganic filler, per cent	41.4	50.2	52.9	51.0	42.1	42.2	49.6	46.1
Impact value at 77 F., ft-lb.	32.0	41.0	30.5	40.5	37.2	43.0	34.0	35.2
Nailing test at 32 F., per cent passable[a]	0	25	50	25	100	100	0	0

[a] To ensure greater uniformity of conditions in the nailing test, the wire nails were driven perpendicularly into the plank by a 10-lb. weight using a modified Johns-Manville impact machine, instead of the specified 6 to 8-lb. hammers.

Materials:

The samples of asphalt plank used in this investigation were made by three of the principal producers of the material and were obtained in the fall of 1936. Asphalt plank represented by samples A, B and C is being sold as bridge flooring, while that represented by D is a harder product sold for use as a railroad ballast waterproofing membrane. Table I describes these samples in terms of composition, resistance to impact and percentage of specimens passing the nailing test.[2]

METHODS OF TESTING

Compression Shear:

The compression-shear tests were run on an Amsler hydraulic testing machine of 20,000-lb. capacity, using the 10,000-lb. setting. A constant temperature bath, 13 by 13 by 5 in., provided with a low-speed electric stirrer and a thermo-regulator controlling an immersion heater provided means for keeping the temperature constant during the tests; at 77 F. and at 125 F. the tem-temperature variation was kept within ± 1 deg. Fahr.; at 32 F. the maximum variation was 3 deg. Fahr., which was always positive. Two Ames dials to measure the deformations were attached to the stationary head of the testing machine, with their plungers resting on the movable head; a flat-faced machined steel plunger, 2 by 2 in. in cross-section, 5 in. long, having its upper end a spherical segment, was used to transmit the load to the surface of the sample.

The sample, 6 by 6 in. by the thickness of the original plank, was held at the required temperature for 2 hr.; it was then placed in the center of the bottom of the constant temperature bath, itself centered on the movable head of the testing machine. The square steel plunger, 2 by 2 in., was centered on the sample and the pressure applied until a total load of 25 lb. was registered on the Amsler dial (6.25 lb.

per sq. in.); this was done to eliminate any roughness which might exist on the surface under test. The Ames dials were then set at zero. The load was increased at the rate of 200 lb. per min. (50 lb. per sq. in. per min.) and the average reading of the dials taken at the end of each minute until a total load of 2800 lb. was reached for the tests at 32 F. and at 77 F. and a total load of 1400 lb. for the tests at 125 F. Each test was run in duplicate. The average indentation in mils was plotted against the load in pounds per square inch.

TABLE II.—DATA FROM COMPRESSION SHEAR TESTS.

Sample	Indentation, mils							
	100 lb. per sq. in.	200 lb. per sq. in.	300 lb. per sq. in.	350 lb. per sq. in.	400 lb. per sq. in.	500 lb. per sq. in.	600 lb. per sq. in.	700 lb. per sq. in.
32 F. (0 C.)								
A-1	25	32	37	39	41	46	51	56
A-2	18	28	35	38	41	47	53	59
B-1	17	25	32	36	40	49	59	69
B-2	25	37	45	48	51	58	65	71
C-1	26	36	43	47	51	59	67	74
C-2	21	34	46	52	58	69	80	91
D-1	15	21	25	27	29	34	38	42
D-2	18	27	33	35	37	42	46	49
77 F. (25 C.)								
A-1	9	25	42	53	65	96	141	208
A-2	15	35	58	71	84	116	152	194
B-1	37	59	93	114	142	223	....	....
B-2	26	38	52	58	65	82	102	127
C-1	27	45	63	74	84	104	129	160
C-2	25	44	68	82	95	124	156	194
D-1	22	29	36	39	43	52	61	72
D-2	23	32	39	43	46	55	65	78
125 F. (52 C.)								
A-1	44	113	234	308	....	....	....	....
A-2	59	151	275	343	....	....	....	....
B-1	71	197	275	315	....	....	....	....
B-2	41	82	128	157	....	....	....	....
C-1	56	102	157	193	....	....	....	....
C-2	75	144	218	260	....	....	....	....
D-1	27	48	72	87	....	....	....	....
D-2	29	52	79	92	....	....	....	....

McBurney Indentation:

The portable instrument described and illustrated in the paper by McBurney[3] was used in the constant temperature bath described above. This instrument delivers a load of 30 lb. made up of an initial load of 2 lb. and an increment of 28 lb. Three diameters of spherical indenting tools, ¼, ½ and 1 in., respectively, were used.

The sample, 6 by 6 in. by the thickness of the original plank was held at the required temperature for 2 hr.; it was then transferred to the leveled constant temperature bath and the instrument placed near the center of one edge, the stop clock started at 10 sec. before zero and the nut rotated until the sphere just touched the surface of the sample. The Ames dial was quickly set to zero and the 28-lb. load released as the clock hand passed the zero mark. After the indenting sphere was fully loaded, readings of the Ames dials were taken after 10, 30, 60, 120, 300 and 600 sec. Three determinations were made at widely separated points on the surface of each sample. The average indentation in mils was plotted against time in seconds on logarithmic paper. The values for a in the McBurney equation:[4] $i = at^m$, were obtained by extrapolating the curves to 1 sec. The values of m were obtained directly from the plotted curves by dividing the distance between the identation at 7 sec. and that at 4000 sec.[5] by the distance between those points on the abscissa. This method was checked against that using the logarithms of the values and substituting in the equation and found to give the same results; being shorter it was adopted for all the calculations.

TEST RESULTS

Table II shows the average results of compression shear test at 32, 77 and 125 F. Figure 1 shows these results graphically. In Table III are given the

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[4] For a description of these terms, see p. 536.

[5] Keuffel & Esser logarithmic paper, 2¾ by 2 cycles.

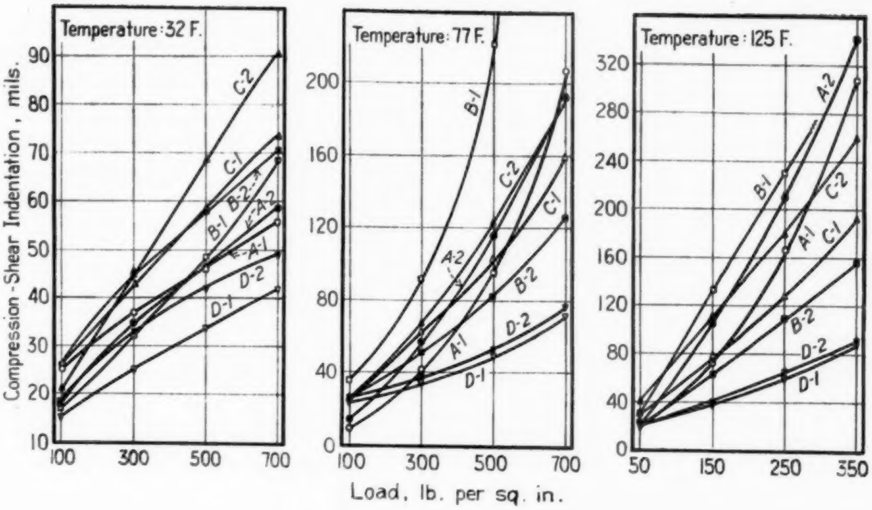

FIG. 1.—Compression-Shear Indentation Curves.

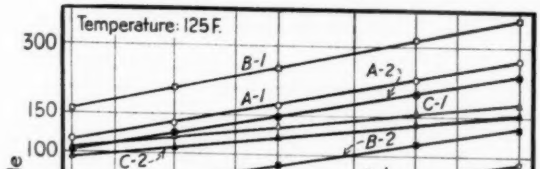

FIG. 2.—McBurney Indentation Curves.

TABLE III.—CONDENSED RESULTS OF INDENTATION TESTS WITH ¼, ½ AND 1-IN. SPHERICAL INDENTERS, 30-lb. WEIGHT.

Sample	¼-in. Indenter			½-in. Indenter			1-in. Indenter		
	a	*m*	Indentation at 600 sec., mils	*a*	*m*	Indentation at 600 sec., mils	*a*	*m*	Indentation at 600 sec., mils
				32 F. (0 C.)					
A-1	7.4	0.162	20.4	3.9	0.175	11.7	....	....	....
A-2	9.6	0.176	29.4	2.6	0.223	10.6	....	....	....
B-1	9.9	0.170	28.9	3.5	0.236	15.1	....	....	....
B-2	11.8	0.100	21.9	5.0	0.135	11.7	....	....	....
C-1	9.3	0.135	21.7	7.4	0.140	17.8	....	....	....
C-2	8.8	0.143	21.9	6.5	0.143	15.7	....	....	....
D-1	4.8	0.128	10.8	1.5	0.154	4.2	....	....	....
D-2	8.1	0.086	13.7	1.6	0.162	4.6	....	....	....
				77 F. (25 C.)					
A-1	20.1	0.246	97.0	15.5	0.197	54.2	10.9	0.202	39.7
A-2	14.9	0.222	61.1	10.5	0.207	39.6	8.7	0.190	29.5
B-1	19.8	0.277	114.4	16.4	0.224	68.0	11.3	0.195	39.6
B-2	16.2	0.174	49.1	10.7	0.168	30.8	10.4	0.125	23.1
C-1	23.8	0.175	80.6	15.2	0.157	47.2	11.9	0.146	33.9
C-2	22.4	0.185	80.8	17.6	0.148	50.6	12.9	0.145	36.9
D-1	7.7	0.197	27.3	5.3	0.194	18.2	4.6	0.165	13.2
D-2	9.7	0.164	26.8	5.5	0.179	16.8	5.1	0.153	12.4
				125 F. (52 C.)					
A-1	....	....	....	43.3	0.281	261.0	32.5	0.249	160.0
A-2	....	....	....	41.8	0.260	225.5	34.6	0.228	137.3
B-1	....	....	....	53.0	0.313	390.0[a]	55.0	0.216	217.0
B-2	....	....	....	29.0	0.238	131.7	27.1	0.194	92.5
C-1	....	....	....	61.3	0.137	169.0	41.0	0.119	98.1
C-2	....	....	....	57.0	0.136	151.6	46.3	0.116	108.6
D-1	....	....	....	21.2	0.232	92.7	18.2	0.207	67.4
D-2	....	....	....	19.3	0.229	81.7	14.8	0.222	60.1

[a] Extrapolated.
Maximum deviation of recorded points from straight line relation was 3.0 per cent.

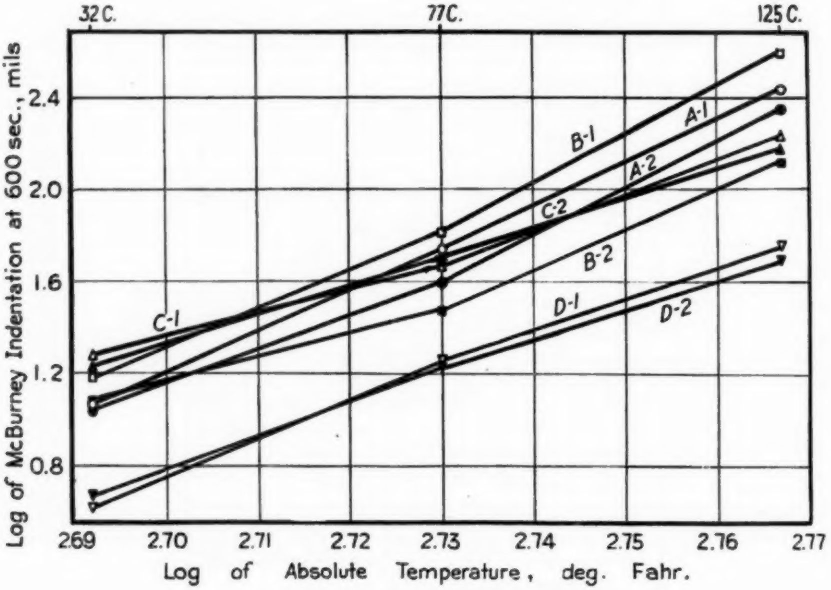

FIG. 3.—Indentation-Temperature Curves.

	SAMPLE A-1	SAMPLE A-2	SAMPLE B-1	SAMPLE B-2	SAMPLE C-1	SAMPLE C-2	SAMPLE D-1	SAMPLE D-2
32 to 77 F.	17.5	14.8	17.2	11.1	11.2	13.4	16.8	14.8
77 to 125 F.	18.4	20.3	20.5	17.1	14.9	12.9	19.0	18.5
32 to 125 F.	18.0	17.5	18.8	14.0	13.0	13.1	17.9	16.6

average results of the indentation tests, using the ¼, ½ and 1-in. spherical indenters, at the same temperatures; Fig. 2 shows these results for the ½-in. indenting tool plotted on a logarithmic scale. It will be noted that no results at 125 F. appear in Table III for the ¼-in. plunger, because indentations higher than 0.250 in., the maximum travel of the plunger, were obtained with most of the samples. With the 1-in. plunger, readings at 32 F. were so small as to be meaningless. Therefore, the ½-in. plunger was chosen to compare indentations given by the McBurney instrument with those obtained under compression shear on similar materials, at the three temperatures, as shown in Fig. 5. In order to measure the temperature susceptibility of the asphalt plank the rates of indentation in the two methods, and relative indentations at the several temperatures were computed as indicated in Tables IV and V based upon the data in Tables II and III, respectively. A graphical method for showing the temperature susceptibility was devised by plotting in Fig. 3 the logarithms of McBurney indentations at 600 sec. against the logarithms of the absolute temperatures in degrees Fahrenheit, taken from Table III. Figure 4, plotted from the data in Table III,

Table IV.—Temperature Susceptibility Factors from Compression Shear Indentations, 350 lb. per sq. in.

Sample	Indentation Increase, mils			Average Indention Increase, mils per deg. Fahr.			$\frac{B}{A}$	$\frac{\text{Indentation at 125 F.}}{\text{Indentation at 77 F.}}$	$\frac{\text{Indentation at 125 F.}}{\text{Indentation at 32 F.}}$
	32 to 77 F.	77 to 125 F.	32 to 125 F.	32 to 77 F.	77 to 125 F.	32 to 125 F.			
				A	*B*	*C*			
A-1	14	255	269	0.31	5.31	2.89	17.1	5.81	7.9
A-2	33	272	305	0.73	5.66	3.28	7.8	4.83	9.0
B-1	78	201	279	1.73	4.18	3.00	2.4	2.77	8.8
B-2	10	99	109	0.22	2.06	1.17	9.4	2.71	3.3
C-1	27	119	146	0.60	2.48	1.57	4.1	2.61	4.1
C-2	30	178	208	0.68	3.71	2.24	5.5	3.17	5.0
D-1	12	48	60	0.27	1.00	0.65	3.7	2.18	3.2
D-2	8	49	57	0.18	1.02	0.61	5.7	2.18	2.6

Table V.—Temperature Susceptibility Factors from McBurney Indentations, 600 sec.

Sample	Indentation Increase, mils			Average Indentation Increase, mils per deg Fahr.			$\frac{B}{A}$	$\frac{\text{Indentation at 125 F.}}{\text{Indentation at 77 F.}}$	$\frac{\text{Indentation at 125 F.}}{\text{Indentation at 32 F.}}$
	32 to 77 F.	77 to 125 F.	32 to 125 F.	32 to 77 F.	77 to 125 F.	32 to 125 F.			
				A	*B*	*C*			
A-1	42.5	206.8	249.3	0.94	4.31	2.68	4.6	4.82	22.3
A-2	29.0	185.9	214.9	0.64	3.87	2.31	6.0	5.69	21.3
B-1	52.9	322.0	374.8	1.18	6.71	4.03	5.7	5.74	25.8
B-2	19.1	100.9	120.0	0.42	2.10	1.29	5.0	4.27	11.3
C-1	29.4	121.8	151.2	0.65	2.54	1.63	3.9	3.58	9.5
C-2	38.6	97.3	135.9	0.86	2.02	1.46	2.3	2.79	9.7
D-1	14.0	74.5	88.5	0.31	1.55	0.95	5.0	5.09	22.1
D-2	12.2	64.9	77.1	0.27	1.35	0.83	5.0	4.86	17.8

shows graphically the relationship between the McBurney values of *m* and temperature for all the samples tested, using the ½-in. plunger.

In order to show more clearly the similarity of results obtained by the two indentation methods, the indentations under compression shear at 350 lb. per sq. in. and the McBurney indentations at 600 sec. were both plotted against temperature in Fig. 5.

DISCUSSION OF TEST DATA

The compression shear curves given in Fig. 1 are practically all straight lines at 32 F., while at 77 F. and at 125 F. they show a tendency to become parabolas. The indentations plotted against time in Fig. 2 show that all the planks tested follow the equation set up by McBurney for asphalt tile:

$$i = at^m$$

where i = the indentation in mils,
t = the time in seconds,
a = the indentation in mils at one second, and
m = the rate at which indentation increases with time.

Hence, it is believed that this equation correctly expresses the stability of plank at a given temperature.

In considering the application of the McBurney equation to stability determinations on asphalt plank it is worth emphasizing that although the value of *m* is of major importance because of its exponential use in the equation, the value *a*, used as a multiplier, also has an important effect. Plank may have a relatively low *m* value and yet indent excessively if the *a* figure is too high.

Because asphalt bridge flooring plank is continuously exposed to the weather and has to withstand traffic at both low and high temperatures, temperature susceptibility is one of its most important properties. As the general shape of the curves in Fig. 5 was seen to be parabolic, when indentations at a specified time, both by the compression shear test and by the McBurney indentation method, were plotted against temperature, it was thought that a definite value might be established for each asphalt plank tested, which would give its temperature susceptibility from the indentation data.

By plotting the logarithms of the McBurney indentations against the logarithms of the absolute temperatures,

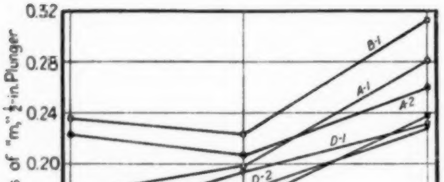

FIG. 4.—Relation of *m* Values to Temperature.

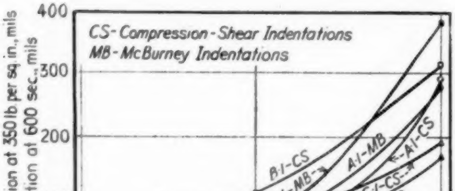

FIG. 5.—Indentation-Temperature Curves.

Fig. 3 was obtained; it shows practically linear relationship for all samples, hence the following equation applies with approximate accuracy:

$$\log i = c + k \log T, \text{ or}$$
$$i = cT^k$$

where i = the indentation in mils at a specified time,

T = the absolute temperature in degrees Fahrenheit,

c = a constant dependent on the material and the load on the indenting tool at the fixed time, and

k = the rate at which indentation increases with temperature, or the temperature susceptibility index.

The calculated values of k given in Fig. 3 differ somewhat between the low-temperature range, 32 F. to 77 F., and the high-temperature range, 77 F. to 125 F.

The different results obtained by the two methods of indentation testing are due to a basic difference in rate of load application. Under compression shear, the pressure *increases* at a uniform rate regardless of the hardness of the material, while in the McBurney test the pressure *decreases* at a rate inversely proportional to the hardness of the material under test; consequently the softer the material, the greater will be the difference between compression shear and McBurney indentations.

Table IV gives data on the indentation increases under compression shear in the low-temperature range, 32 to 77 F., in comparison with those in the high-temperature range, 77 F. to 125 F. The total indentation increase in the high-temperature range is particularly useful in predicting whether the plank will become too soft in hot weather, assuming the indentation at 77 F. indicates satisfactory stability at that temperature. Various ratios may be used to express temperature susceptibility, using compression-shear data, and values from three such ratios are given in the table. The values obtained indicate the importance of considering stability and temperature susceptibility *together*, since a plank may have a relatively low susceptibility factor and yet be of unsatisfactory stability, as an example, sample B-1. It should be noted that planks of differing thickness, although made of the same composition, will show different indentation behavior and the 1½ in. thick samples, A-2 and C-2 illustrate this, showing higher indentations than A-1 and C-1, respectively.

In Table V similar data on indentation increases in the two temperature ranges, obtained with the McBurney instrument, are given, together with three ratios expressing temperature susceptibility, similar to those presented in Table IV. From the results it appears that the ratio of the 600-sec. indentations at 125 F. and 77 F. affords a satisfactory index of temperature susceptibility, requiring only two determinations.

When the values of m, the constant which expresses the tendency to indent with time, are plotted against temperature in Fig. 4, samples C-1 and C-2 show the least change in numerical value over the temperature range of tests, while all the other samples show a comparatively large increase between 77 F. and 125 F. That this difference in behavior between C-1 and C-2 and all the other samples is one of structure is forcibly brought out by the fact that on the ash determination for analysis, when all the organic matter is burned off, the original size and shape of the plank samples are retained in the case of C-1 and C-2, while the other samples melt to the bottom of the crucible before burning off. This greater stability due to structure is also shown by the low-temperature susceptibility of those two samples (Fig. 3), by their relatively constant values of m up to 125 F., by their greater toughness under the impact test, and by their ability to withstand nailing at 32 F. without cracking.

Samples D-1 and D-2 were included in this series of asphalt planks to show that the susceptibility factor is not the only criterion to be considered in the physical properties of this product, but that the hardness at each temperature is also of primary importance. The ideal material for bridge surfacing would of course be one which would have the same hardness at 32 F. as at 125 F. Asphalt planks, such as D-1 and D-2, which are extremely hard at 77 F. tend to be much too hard at 32 F. and consequently brittle at that temperature; this hardness is shown by the low indentations recorded for these two samples both under compression shear and under the McBurney indenter at 32 F. The brittleness is shown by the results of the nailing tests given in Table I; this test, which may be criticized because it lacks a scientific basis, is nevertheless of great value to the user of asphalt plank who requires a plank that can be nailed to the bridge subfloor either in winter or in summer. On the other hand too soft an asphalt plank, like B-1, will split under compression shear both at 77 F. and at 125 F. and will also show an abnormally high McBurney indentation at 125 F.; in service it will rut easily.

The dynamic impact value of asphalt plank at 77 F. is also considered an important property by the authors, especially since a large number of asphalt plank installations are made over timber sub-floors which move appreciably under the impact of heavy cars and trucks, causing the asphalt plank nailed and cemented to them to move with them. The impact test has been found to be an excellent criterion of the toughness of asphalt plank under repeated blows.

In writing specifications for asphalt plank, it is suggested that at least one physical test at each of three temperatures should be required; at 77 F. and at 125 F. indentation tests should be made, either by the compression shear method or with the McBurney instrument, defining maximum limits for each indentation and a maximum limit on the ratio of the indentation at 125 F. to that at 77 F. At 32 F. either indentation method might be used, setting a minimum limit to avoid unduly high hardness or specifying the nailing test to insure against brittleness at that temperature. An impact test at 77 F. should also be included to insure against failure under impact stresses in service.

A large number of bridges surfaced with asphalt plank was tested with the McBurney instrument, using the ¼-in. spherical plunger at temperatures varying from 60 F. to 80 F., but the data obtained were not considered accurate enough to include in this paper, because of the large temperature variations found between the surface of the planks and points ¼ to ½ in. beneath the surface. The results of these determinations, while giving a fair indication of the hardness of the planking material by its indentation at 600 sec. and its rate of indentation increase with time, did not give any information on temperature susceptibility. Another difficulty encountered after the field data were compiled was the lack of a definite relationship between the indentation at 600 sec. or the value of m and the size of the indenting tool.

A study of Table I in conjunction with all the indentation data recorded for the eight samples will show a complete lack of relationship between composition and physical properties, because of the different types of asphalt, mineral fillers and organic fibers used by each manufacturer of asphalt plank; for this reason it is believed that asphalt plank specifications should be based only on the physical properties of the product and not on its composition.

SUMMARY

1. Two types of indentation test have been shown to be satisfactory for determining the stability of asphalt plank: the compression shear method and the McBurney indentation method.

2. It has been found that the equation developed by McBurney for asphalt tile: $i = at^m$ applies also to all the asphalt planks tested, at the three temperatures investigated.

3. Methods of determining the temperature susceptibility of asphalt plank have been devised, both mathematically and empirically, by using data from either indentation test.

4. The relation between absolute temperature in degrees Fahrenheit and the McBurney indentation in mils after a specified time is expressed by the equation $i = cT^k$.

5. Supplementary test methods have been recommended for inclusion in purchase specifications for asphalt plank.

6. It has been demonstrated that the physical properties, particularly stability, of one type of asphalt plank tested are associated with its rigid structure.

7. It has been shown that, for all eight samples tested, no relationship exists between composition and physical properties.

DISCUSSION

Mr. J. W. McBurney[1] (*presented in written form*).—The writer understands the feelings of the mother who left her baby on the stranger's door step and later sees and recognizes her child now walking and talking. Like the mother's, most of my comments are questions on the childs upbringing and conduct:

1. Working with $\frac{3}{16}$-in. asphalt tile, some rather definite evidence was obtained which indicated that the relation $i = at^m$ failed to hold when the depth of indentation i exceeded one-half of the radius of the indenting sphere. It was recommended that the maximum depth of indentation be limited to one-third the radius if any considerable accuracy in the determination of m is desired. May not this, in part, explain the differences in the m when $\frac{1}{4}$ and 1-in. spheres are compared? With nothing more than a "hunch" as reason, replacement of the sphere by a paraboloid of revolution had been suggested. It is the writer's understanding that this works.

2. The paper compares indentation in mils at 350 lb. per sq. in. for the compression shear test with indentation in mils at 600 sec. for the sphere. Since the compression shear test involves a uniform rate of loading, would it not be possible to express the results as a rate of indentation corresponding to the rates calculated by differentiating the $i = at^m$ relation? Plotting these rates against pounds per square inch as was done in a previous paper,[2] would provide a more direct comparison of the two methods.

3. In the discussion of specification requirements it is not clear whether it is intended to use the 600-sec. indentation alone or take account of the constant m. Ten-minute indentation is a better basis for comparison than is one minute but a comparison based on 24-hr. or 1-yr. indentation is still better.

4. The McBurney-Bowen indentation tester has its faults. Among these are the use of a considerable load (2 lb.) for the initial load. It would be very much nicer from the standpoint of theory if the zero reading corresponded to zero load. Release of the 28-lb. load involves some impact. Whittemore[3] in 1935 published some results on clay testing. He took elaborate precautions to insure zero load at zero indentation, and release of load without impact. His m's reproduced to ± 0.0001. Whittemore's first method consisted in using an increasing load on a flat ended pin. His final and recommended method involved a constant load acting on a sphere.

Mr. D. T. Colton[4] (*presented in written form*).—The conclusions of Greider and Marc with regard to the application of the McBurney equation to asphalt plank agree closely with results

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[1] Senior Technologist, National Bureau of Standards, Washington, D. C.

[2] J. W. McBurney, "Indentation of Asphalt Tile," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, Fig. 4, p. 599 (1934).

[3] J. W. Whittemore, "Mechanical Methods for the Measurement of the Plasticity of Clays and Mixtures of Clay," *Journal*, Am. Ceramic Soc., Vol. 14, No. 11, pp. 352–359 (1935).

[4] Research Engineer, Johns-Manville Research Laboratories, Manville, N. J.

which have been obtained at this laboratory. In this latter work, two types of plank as manufactured by three producers were examined. Plain and mineral surfaced specimens were tested in three common thicknesses. Data are presented for McBurney tests made on asphalt plank at temperatures of 32, 77, 115 and 140 F.

TABLE I.—AVERAGE INDENTATION DATA ON PLAIN ASPHALT PLANK.

McBurney-Bowen indentation tester with 1-in. indenter.

Sample	Thickness, in.	Indentation, mils			*m*	*a*
		30 sec.	60 sec.	600 sec.		
		32 F.				
X	1	......	11.4	17.2	0.179	5.3
	1¼	......	10.9	16.6	0.182	5.0
	1½	......	10.5	15.2	0.161	5.4
Y	1	......	6.0	8.4	0.146	3.3
	1¼	......	6.0	8.8	0.166	3.1
	1½	......	5.6	7.8	0.144	3.1
Z	1	......	6.1	8.4	0.139	3.4
	1¼	......	6.6	9.1	0.140	3.7
	1½	......	6.8	9.2	0.131	3.9
		77 F.				
X	1	......	39.2	59.2	0.179	18.9
	1¼	......	34.0	49.2	0.160	17.5
	1½	......	30.3	46.3	0.184	14.3
Y	1	......	21.9	33.0	0.179	10.5
	1¼	......	18.1	25.9	0.155	9.8
	1½	......	16.1	24.9	0.189	7.5
Z	1	......	17.7	25.7	0.162	9.1
	1¼	......	16.7	24.8	0.172	8.2
	1½	......	18.0	26.6	0.170	9.1
		115 F.		(300 sec.)		
X	1	100.3	115.7	162.3	0.209	51
	1¼	100.0	116.0	161.0	0.207	49
	1½	90.3	105.5	146.7	0.210	44
Y	1	55.5	63.0	83.2	0.176	31
	1¼	52.5	61.2	82.0	0.194	27
	1½	47.8	54.8	73.3	0.186	25
Z	1	50.7	59.0	82.7	0.212	24
	1¼	48.5	56.8	81.5	0.225	23
	1½	51.5	59.8	85.3	0.220	24
		140 F.				
X	1	221.2	......	......	......	......
	1¼	193.7	263.3	......	0.443	......
	1½	173.7	200.6	......	0.205	83
Y	1	97.4	109.9	......	0.175	55
	1¼	101.8	114.3	......	0.166	59
	1½	84.1	92.8	......	0.138	52
Z	1	94.2	107.7	......	0.193	48
	1¼	89.7	104.2	......	0.218	43
	1½	90.8	105.8	......	0.220	44

The McBurney-Bowen Indentation Tester was employed in making this study. All tests were conducted in a large water bath. Circulation and automatic temperature regulation were provided as well as storage space for specimens during conditioning.

TABLE II.—AVERAGE INDENTATION DATA ON MINERAL SURFACED ASPHALT PLANK.

McBurney-Bowen indentation tester with 1-in. indenter.

Sample	Thickness, in.	Indentation, mils			*m*	*a*
		30 sec.	60 sec.	600 sec.		
		32 F.				
X-1	1	......	12.2	17.8	0.164	6.2
	1¼	......	10.9	16.1	0.170	5.3
	1½	......	11.5	16.5	0.156	6.1
Y-1	1	......	4.4	6.5	0.169	2.2
	1¼	......	4.2	6.1	0.162	2.1
	1½	......	4.8	6.8	0.151	2.6
Z-1	1	......	6.3	8.4	0.125	3.8
	1¼		6.5	8.9	0.136	3.7
	1½	......	6.1	8.8	0.159	3.2
		77 F.				
X-1	1	......	41.4	64.7	0.194	18.8
	1¼	......	37.5	57.7	0.187	17.5
	1½	......	37.1	55.5	0.175	18.3
Y-1	1	......	14.0	20.5	0.165	7.1
	1¼	......	16.0	25.0	0.193	7.2
	1½	......	14.6	21.2	0.162	7.5
Z-1	1	......	18.2	27.7	0.182	8.6
	1¼	......	18.5	27.8	0.176	8.9
	1½	......	18.4	25.9	0.148	10.0
		115 F.		(300 sec.)		
X-1	1	118.3	137.7	193.0	0.212	57
	1¼	108.3	128.3	182.2	0.225	50
	1½	87.2	102.5	147.2	0.228	40
Y-1	1	50.7	57.7	75.5	0.173	28
	1¼	45.0	51.8	70.7	0.196	22
	1½	46.7	53.2	69.2	0.170	26
Z-1	1	45.7	52.3	73.0	0.204	23
	1¼	48.3	56.0	78.0	0.209	24
	1½	46.7	54.7	77.0	0.217	22
		140 F.				
X-1	1	196.5	239.5	......	0.286	77
	1¼	193.0	227.5	......	0.237	86
	1½	174.7	202.9	......	0.214	81
Y-1	1	85.8	96.0	......	0.161	48
	1¼	87.2	97.6	......	0.161	50
	1½	78.6	85.6	......	0.122	52
Z-1	1	96.8	110.6	......	0.193	51
	1¼	93.8	106.9	......	0.188	48
	1½	95.9	110.9	......	0.209	45

The indentation procedure closely paralleled that described in the paper except that readings of indentation were taken at different time periods. All specimens were tested at four temperatures, samples being conditioned for not less than two nor more than three hours prior to test. A 1-in. indenter was used in the McBurney machine. All tests were performed by the same operator. Indenting periods were chosen to give suitable readings without exceeding the capacity of the indenter.

TABLE III.—AVERAGE INDENTATION DATA ON ASPHALT PLANK AT 115 F.

McBurney-Bowen indentation tester with 1-in. indenter.

Sample	Thickness, in.	Indentation, mils				Percentage Deviation of Computed from Observed Indentations
		30 sec.	60 sec.	300 sec.		
				Computed	Observed	
			PLAIN			
X	1	100.3	115.7	163.0	162.3	+0.4
	$1\frac{1}{4}$	100.0	116.0	159.6	161.0	−0.9
	$1\frac{1}{2}$	90.3	105.5	147.2	146.7	+0.3
Y	1	55.5	63.0	84.5	83.2	+1.6
	$1\frac{1}{4}$	52.5	61.2	81.7	82.0	−0.4
	$1\frac{1}{2}$	47.8	54.8	72.3	73.3	−1.4
Z	1	50.7	59.0	81.8	82.7	−1.1
	$1\frac{1}{4}$	48.5	56.8	81.1	81.5	−0.5
	$1\frac{1}{2}$	51.5	59.8	85.7	85.3	+0.5
			MINERAL SURFACED			
X-1	1	118.3	137.7	190.6	193.0	−1.2
	$1\frac{1}{4}$	108.3	128.3	180.3	182.2	−1.0
	$1\frac{1}{2}$	87.2	102.5	146.6	147.2	−0.4
Y-1	1	50.7	57.7	75.0	75.5	−0.7
	$1\frac{1}{4}$	45.0	51.8	68.7	70.7	−2.8
	$1\frac{1}{2}$	46.7	53.2	68.6	69.2	−0.9
Z-1	1	45.7	52.3	72.0	73.0	−1.4
	$1\frac{1}{4}$	48.3	56.0	78.9	78.0	+1.2
	$1\frac{1}{2}$	46.7	54.7	75.9	77.0	−1.4

The choice of a 1-in. indenter was made as a result of an exhaustive series of tests using $\frac{1}{4}$, $\frac{1}{2}$ and 1-in. indenters. It was found that certain plank showed indentations too high for the smaller indenters at temperatures of 115 F. and above. Discrepancies which may be observed in the Greider and Marc data due to the choice of too small an indenter are discussed later.

Values of a were obtained by extrapolation from curves of indentation against time as plotted on log-log paper. Values of m were computed. Properly chosen indentation periods simplify the computation of m values.

In McBurney's equation $i = at^m$, it can be shown that

$$m = \log\left(\frac{i_1}{i_2}\right);\ \text{if}\ \log\left(\frac{t_1}{t_2}\right) = 1$$

thus, if time periods of 600 and 60 sec. or 300 and 30 sec. are chosen, m values may be found rapidly and accurately by means of a slide rule.

Typical specimens of each type of plank were analyzed. Percentage composition and characteristics of recovered bitumen were obtained. Small patties $\frac{3}{16}$ in. thick were made from the asphalt recovered and subjected to indentation tests with the McBurney machine at 77 F. (1-in. indenter).

Field indentations on asphalt plank installed on bridge decks were made with the McBurney-Bowen indentation tester. Temperatures were measured with black bulb thermometers. Standard samples of plank were tested in the laboratory and then taken into the field and tested under conditions prevailing on each bridge.

Average indentation data for plain asphalt plank are given in the accompanying Table I. Values of m and a are included. Corresponding data for mineral surfaced plank are found in the accompanying Table II. Each indentation value in Tables I and II represents the average of at least nine individual readings, equally divided among three specimens.

In order to show the close check between actual and computed indentation values when proper size of indenter is employed, Table III was drawn up from portions of Tables I and II. Data were tabulated for indentations at 115 F.

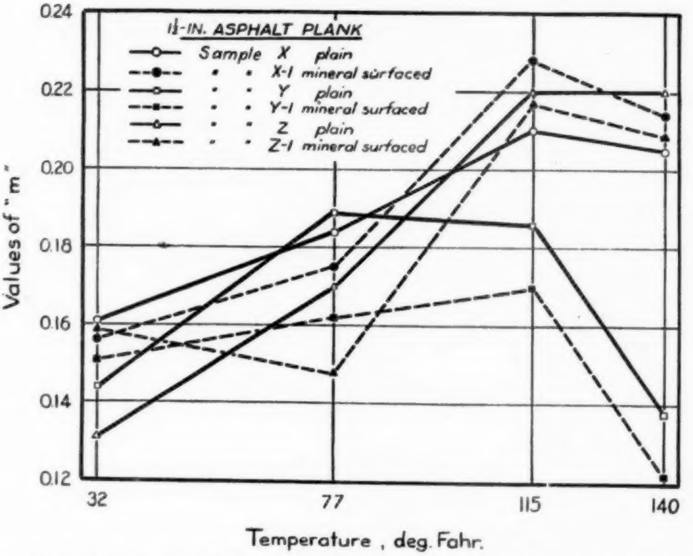

FIG. 1.—Relation of *m* Values to Testing Temperature, 1½-in. Asphalt Plank.

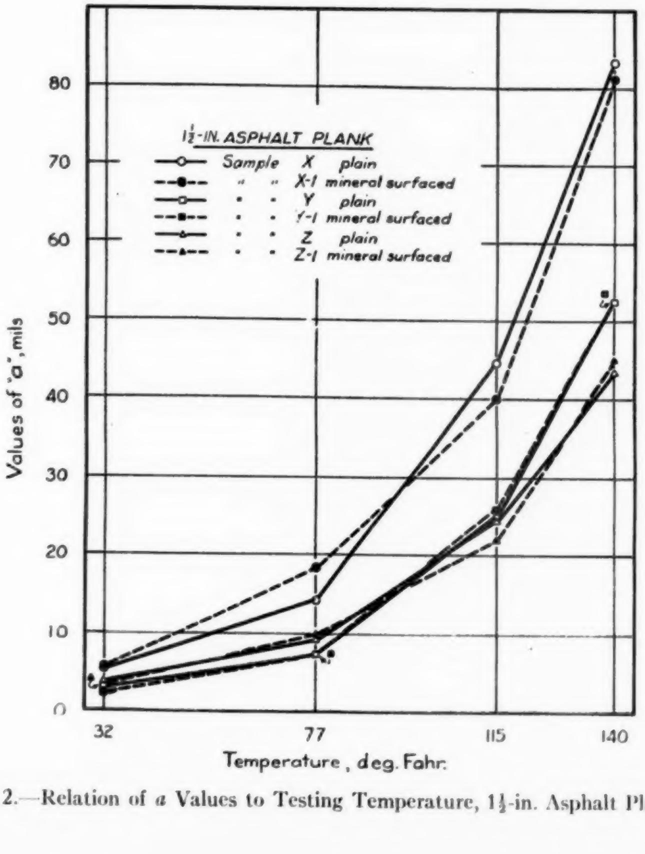

FIG. 2.—Relation of *a* Values to Testing Temperature, 1½-in. Asphalt Plank.

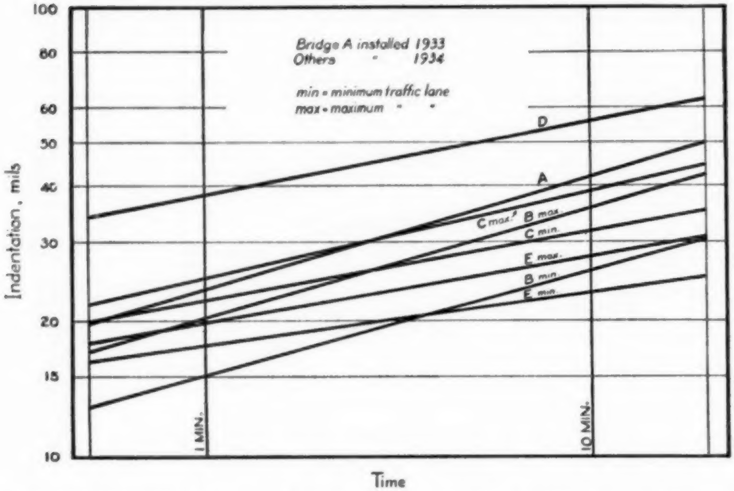

FIG. 3.—Time-Indentation Curves—Asphalt Plank. Typical bridge installations. McBurney Bowen indentation tester—1-in. indenter.

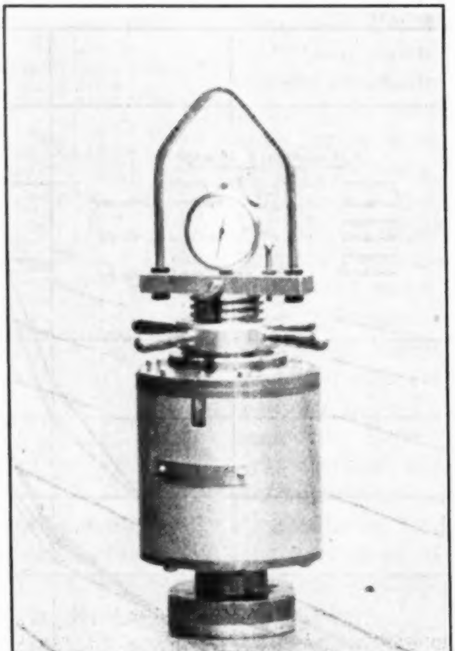

FIG. 4.—McBurney Machine in Use on Asphalt Plank Bridge Surface.

Indentations at 300 sec. were computed from 30 and 60-sec. values. Differences between actual and computed indentations are shown as percentages of the observed figures.

In Table IV, typical percentage compositions of the various plank specimens are shown. Melting point, penetration and McBurney indentation values are given for the recovered bitumen.

The accompanying Fig. 1 shows values of m plotted against temperature for plain and mineral surfaced plank. Data for these curves are those in Tables I and II for $1\frac{1}{2}$-in. plank. Similarly, the accompanying Fig. 2 shows a values plotted against temperature for $1\frac{1}{2}$-in. asphalt plank.

The accompanying Fig. 3 shows graphically the results of a series of indentation tests on six bridges. Where available, indentation data are given for maximum and minimum traffic lanes on a bridge. All results are calculated to a temperature of 77 F.

The accompanying Fig. 4 shows an indentation test in progress on bridge plank in the field.

Greider and Marc have confirmed our conclusions of a year and one-half ago regarding the applicability of McBurney's equation $i = at^m$ to asphalt plank indentations. A definite indication of the accuracy with which this equation may be used to compute indentations is found in the accompanying Table III. The extremely low percentage deviation between computed and observed indentations is explained by the fact that each line of data represents the average of at least nine readings. Thus, the individual variation is not indicated.

Examination of the data in the accompanying Tables I and II points to some interesting conclusions. Plain and mineral surfaced plank show similar indentation behavior. Variations in plank thickness apparently may cause more pronounced changes in indentations than alteration of surfacing. However, these effects vary with the source of the material. Plank represented by group X tend to be somewhat more resistant to penetration in greater thicknesses. Plank groups Y and Z do not show any consistent changes with thickness.

In general, group X represents a plank material which is poorly suited for use as a bridge surfacing. Groups Y and Z show good resistance to indentation at all temperatures. Material X, having slightly lower m values would indent less under relatively long time static load than would plank Z. At the higher temperatures, material Z would be less indented by moving loads than material Y.

Due to the large indenting plunger used, relative to thickness of sample the compression-shear test may give false comparisons between plank of different thicknesses. This effect may be observed in sample C-1 and C-2 of the Greider and Marc data. These plank, of identical composition, were 1 and $1\frac{1}{2}$ in. in thickness, respectively. At all three temperatures, the Greider and Marc compression-shear tests indicate that the thinner plank is harder. Their McBurney indentation tests on the same materials show that the 1-in. plank is softer than the $1\frac{1}{2}$-in. material at 32 F. and 125 F. and slightly harder at 77 F.

In applying the McBurney equation to asphalt plank, variation in the exponent m is caused by change in material and temperature. Greider and Marc recognized this fact but also further stated "its value increases rapidly at temperatures above 77 F." Reference to the accompanying Fig. 1 shows m values increasing with temperature to 115 F. and then dropping off when the temperature was further increased to

140 F. This effect was not observed by Greider and Marc at 125 F. although it was indicated by their samples C-1 and C-2. The explanation for this phenomenon lies in the physical structure of the plank. At high temperatures, the bitumen in a plank no longer controls resistance to indentation. This resistance is provided by the fibrous structure or filler, which is not softened by heat. Samples Y and Y-1 exhibit excellent structure; other samples do not. McBurney indentation tests were made at 77 F. on bitumens extracted from various plank samples (accompanying Table IV) but unfortunately could not be made at higher temperatures.

TABLE IV.—COMPOSITION OF ASPHALT PLANK AND CHARACTERISTICS OF RECOVERED BITUMEN.

	SAMPLE X	SAMPLE Y	SAMPLE Z
Percentage composition:			
Bitumen	36.9	39.8	44.9
Mineral matter	47.2	46.1	41.3
Organic fiber	15.9	14.1	13.8
Characteristics of recovered bitumen:			
Ring-and-ball melting point	221 F.	207 F.	228 F.
Penetration—32 F. 200-g. weight, 60 sec.	11	4	4
Penetration—77 F. 100-g. weight, 5 sec.	18	8	8
Penetration—115 F., 50-g. weight, 5 sec	32	16	12
McBurney indentations on bitumen, mils:			
30 sec.—77 F.	85.0	38.0	43.5
60 sec.—77 F.	98.0	44.0	51.0
300 sec.—77 F.	129.0	62.5	71.8
Value of *m*	0.181	0.216	0.217
Value of *a*, mils	46.0	18.2	20.8

McBurney stated that if *m* exceeds 0.20, asphalt tile indent deeply as a rule. Apparently, some similar limit may be set for asphalt plank. Note that *m* values for samples Y and Y-1 are below 0.190 at all temperatures (accompanying Fig. 1).

The accompanying Fig. 2 shows that values of *a* increase rapidly as temperature is raised. In a general way, *a* values remain in corresponding relationship at various temperatures. In asphalt plank, *a* may be considered as a measure of resistance to indentation by traffic (moving loads instantaneously applied).

Several characteristics of indentation must be controlled by a specification which would be considered suitable for asphalt plank. The Greider and Marc susceptibility relationship between indentation and temperature is of theoretical interest only. Computations of susceptibility factors should not be necessary in specification testing. If *m* is controlled by suitable indentation limits for 1- and 10-min. readings at some normal temperature, such as 77 F., and if increase in *a* with temperature is kept to a minimum by a maximum allowable 30-sec. indentation at some higher temperature, such as 115, 125 or 140 F., a specification can be written which would describe plank with optimum indentation resistance.

Reference to Table III of the paper shows that the $\frac{1}{2}$-in. indenting point limit of 125 mils was exceeded in six of the eight measurements at 125 F. This would result in excessive measured indentation and high *m* values.

The accompanying Fig. 3 shows time-indentation curves for six typical bridge installations of asphalt plank. Bridges are lettered in order of approximate traffic density, bridge A having about 1500 cars per hour and bridge E about 25 cars per hour. Approximate parallelism is found between indentation curves in maximum and minimum traffic areas for each structure. Note that maximum traffic area shows higher indentations than does the little travelled portion of the bridge surface in each case. This implies a softening of asphalt plank with service, that is, mechanical working of plank under traffic results in slight indentation increase but no marked changes in *m* values.

These data on bridge installations of asphalt plank are extremely interesting. However, measurements made in the field are subject to many variations. The most important of these are vibration of bridge structure and lack of temperature control. Although the curves in the accompanying Fig. 3 are prepared from scores of individual readings, further work is being performed on samples brought into the laboratory where conditions of test may be controlled.

Messrs. H. W. Greider[5] and Henri Marc[5] (*authors' closure, by letter*).—From a study of their own data, the authors find it difficult to agree with Mr. McBurney's statement that the differences in the *m* value when $\frac{1}{4}$ and 1-in. spheres are compared are primarily due to errors caused by indentations exceeding one-half of the radius of the indenting sphere. The maximum deviation of recorded points from a straight-line relation is only 3.0 per cent and all but two of the curves at 125 F. in Fig. 2 have an indentation under 125 mils at 60 sec., the second point used in the calculation of the straight-line relation, the first being taken at 30 sec. This difference of opinion in regard to the value of *m* will only be settled when a true paraboloid of revolution can be used as the indenting point.

The authors agree with Mr. McBurney that it might be possible to express the comparison between the two indentation methods somewhat differently than was shown in Fig. 5, but they merely wished to show that a general relationship exists between the results obtained by the two methods of indentation testing. In the discussion of specification requirements, 600-sec. indentations at two temperatures are suggested to exclude asphalt planks with abnormally high *a* values as well as those with high *m* exponentials.

The two faults of the indentation tester mentioned by Mr. McBurney were noticed early in the work, especially at the higher temperatures, and for this reason a 10-sec. maximum time of application of the 2-lb. load was strictly adhered to; nothing could be done about the impact produced by the addition of the 28-lb. load. In spite of Mr. McBurney's criticisms of the instrument of his own design, we feel indebted to him for giving us an additional and convenient method of determining the consistency (stability) of industrial plastic materials.

The authors also wish again to give due credit to Mr. Colton who, having used the McBurney indenter in connection with asphalt tile, recognized its possibilities as a testing instrument for asphalt plank. A comparison of the data submitted by Mr. Colton and the authors' data may be misleading, as, of the three types of asphalt plank tested by him, only the sample Y corresponded to sample C in the paper in general structure and manufacturing formulation, the others being entirely different in properties and composition.

The authors consider Mr. Colton's data on the variations of indentation with plank thickness interesting, but beyond the scope of a discussion of the paper. That such variations should exist is not difficult to understand when one considers that the pressure exerted on the planking material as it goes through the extrusion die varies inversely with the cross-section of the individual die, as well as with the die temperature and that of the material before extrusion, regardless of its composition. The difference in relative hardness between samples C-1 and C-2 when tested by both indentation methods was taken by Mr. Colton as being an indication of the shortcomings of the compression shear test. This is

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[5] Director of Research, and Research Chemist, respectively, The Philip Carey Manufacturing Co., Lockland, Ohio.

not true, however, when bottom effects are taken into consideration in conjunction with compression tests on plastic materials. These are not shown in results obtained with the McBurney indenter because of the low pressures to which the material is subjected under that test.

Had the authors chosen to publish all their data on McBurney indentations instead of only those which could be correlated with compression shear results, the decrease in m value at 140 F. for sample C would have been brought out in the paper; observations on this property on material similar to sample C were communicated privately to Mr. Colton in December, 1936.

The authors definitely do not agree with Mr. Colton in his statement that the temperature susceptibility relationship is of theoretical interest only, since all his data, as well as the authors', show that asphalt plank is a thermoplastic material which fails primarily *because of excessive indentation at elevated temperatures*; they are of the opinion that in writing specifications, the limits set at the higher temperature should be computed from those required at the lower temperature, using a reasonable temperature susceptibility factor.

The authors wish to thank Messrs. McBurney and Colton for their thorough study of this paper and hope that its discussion will justify an old French proverb that "from the impact of ideas comes light."

DEVELOPMENT OF INTERNAL STRUCTURE IN ASPHALTS WITH TIME

By R. N. Traxler[1] and C. E. Coombs[2]

Synopsis

The increase in consistency of asphalts with time indicates the development of internal structure. This structure is developed more rapidly in air-blown than in steam- or vacuum-refined bitumens; it is partially or wholly eliminated by heating or mechanical working. In asphalt-mineral mixtures age-hardening appears to be chiefly dependent on the nature of the bitumen present.

It has been indicated that the higher the rate at which internal structure develops, the more marked the deviation from viscous flow.

Asphalts with high rates of age-hardening and definite deviations from viscous flow give characteristic microscopic patterns when the surfaces are etched with ethyl ether or 86° Baumé naphtha. Essentially viscous asphalts have low rates of age hardening and show no surface patterns.

The explanation offered for the increase of consistency with time is that a gradual isothermal sol-gel transformation takes place, the magnitude of which depends on the source and method of processing.

Recently it was shown[3] (1) that the consistencies of asphalts increase with time, (2) that the rate of increase varies with the kind of asphalt, and (3) that unless several thousand hours have elapsed the increase in consistency can be partially or wholly eliminated by heating or mechanical working. In the present paper further data are given and the dependence of the age-hardening of an asphalt on its internal structure is discussed.

Experimental Procedure

Several small-size falling coaxial cylinder viscometers[4] (sample volume = 16 cu. cm.) were filled with the material to be tested and then stored at a constant temperature (25 C., 77 F.). At desired intervals a viscometer was removed from the constant-temperature cabinet and the consistency determined at 25 C. (77 F.).

A simple control viscometer utilizing the principle of the falling coaxial cylinder was recently developed[5]; this apparatus has been used successfully in recent age-hardening studies.

Essentially Viscous Bitumens

The essentially viscous asphalts and bitumens studied have been found to give a linear relationship between log-viscosity and log-time.[3] This is expressed by the equation

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[2] Formerly with The Barber Co., Inc. Present address: Technical Dept., E. I. du Pont de Nemours and Co., Buffalo, N. Y.

[3] R. N. Traxler and H. E. Schweyer, "Increase of Viscosity of Asphalts with Time," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 544 (1936).

[4] R. N. Traxler and H. E. Schweyer, "Measurement of High Viscosity—a Rapid Method," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 518 (1936).

[5] R. N. Traxler, "The Flow Properties of Asphalts Measured in Absolute Units," Symposium on Consistency, Am. Soc. Testing Mats. (1937). (Symposium available as separate publication.)

$$\eta = b\, t^{m} \quad \ldots\ldots\ldots\ldots (1)$$

where η = viscosity in poises at 25 C. (77 F.),

t = time of aging in hours, and

m and b are constants.

Since the rate of increase of viscosity with respect to time is not constant but is a function of the viscosity and the time, the age-hardening has been arbitrarily evaluated by the rate of hardening at 100 hr. This value has been called the Asphalt Aging Index (A.A.I.)[3]

Table I lists in order of increasing A.A.I. the bitumens that have been found to obey Eq. 1.

TABLE I.—PHYSICAL CHARACTERISTICS AND A.A.I. AT 25 C. (77 F.) FOR VARIOUS BITUMENS.

Designation	Source (Type)	Process	Penetration, 77 F., 100-g. weight, 5 sec.	Softening Point, Ring-and-Ball Method, deg. Fahr.	A.A.I. at 25 C. (77 F.)
A.......	Californian	Vacuum	35	122	0.012
B.......	Californian	Vacuum	55	117	0.018
C.......	Coal tar	....	19	130	0.024
D.......	Trinidad oil	Batch steam	55	121	0.026
E.......	Kansas	Pressure still	77	109	0.035
F.......	Venezuelan	Vacuum	57	124	0.037
G.......	Fluxed Refined Trinidad Native Lake	....	56	127	0.038
H.......	Mid-Continent	Vacuum	56	123	0.039
I.......	Venezuelan	Vacuum	167	103	0.039
J.......	Mexican	Vacuum	55	126	0.051
K.......	Venezuelan	Batch steam	63	125	0.063
L.......	Venezuelan	Batch steam	53	128	0.071[a]

[a] This asphalt did not give a straight line on the log-log plot. The A.A.I. value was taken from the best straight line calculated from the experimental data up to 1000 hr.

The oil asphalts listed in Table I would be called homogeneous by the Oliensis Test.[6,7] However, judging from the extent of the anomalous flow properties encountered in these different kinds of asphalt, the internal structure does seem to increase, in a general way, with increasing A.A.I. values. Californian asphalt of 35 to 50 penetration displays practically no anomalous characteristics while Venezuelan vacuum-refined asphalt of 50 penetration shows very slight elastic and quasi-viscous effects; in batch steam-refined Venezuelan asphalt of 50 penetration the flow properties are definitely anomalous. The complexity of the flow properties has been found to be directly related to the degree of colloidality of the asphalt.[8,9]

It was found that in most cases the viscosity of an age-hardened asphalt could be decreased markedly by heat. This was shown when samples which had age hardened for several thousand hours were remelted in the viscometer, allowed to cool, and the viscosity determined as soon as a constant temperature had been attained. Such behavior shows that a large proportion of the increase in consistency of asphalts with time (during the first few thousand hours) is due to a reversible process occurring within these colloidal materials.

NON-VISCOUS ASPHALTS

Age-hardening characteristics were determined on three air-blown asphalts of

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[6] G. L. Oliensis, "A Qualitative Test for Determining the Degree of Heterogeneity of Asphalts," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 715 (1933).

[7] G. L. Oliensis, "A Further Study of the Heterogeneity of Asphalt—a Quantitative Method," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 494 (1936).

[8] R. N. Traxler and C. E. Coombs, "The Colloidal Nature of Asphalt as Shown by Its Flow Properties," *Journal of Physical Chemistry*, Vol. 40, p. 1133 (1936).

[9] J. P. Pfeiffer and P. M. Van Doormaal, "The Rheological Properties of Asphaltic Bitumens," *Journal*, Inst. Petroleum Technologists, Vol. 22, p. 414 (1936).

about the same ring-and-ball softening point. These asphalts, all of which were quasi-viscous (non-Newtonian),[10] were prepared from the same type of Venezuelan petroleum but possessed different characteristics depending upon the extent to which they had been blown. The physical characteristics of these asphalts are recorded in Table II.

Several viscometers were filled with each of these asphalts, the samples aged at 25 C. (77 F.) and determinations made at that temperature at intervals up to 3000 hr. Since these asphalts were non-Newtonian liquids, the same load (mean shearing stress = 4400 dynes per sq. cm.) was used for all of the tests in order that the values for "appar-

TABLE II.—PROPERTIES OF AIR-BLOWN ASPHALTS.

Designation	Blown	Penetrations			Softening Point, Ring-and-Ball Method, deg. Fahr.
		32 F., 200-g. weight, 60 sec.	77 F., 100-g. weight, 5 sec.	115 F., 50-g. weight, 5 sec.	
M....	Slightly	16	54	270	133
N....	Moderately	34	87	......	127
O....	Highly	53	122	......	124

TABLE III.—DATA FOR AGE-HARDENING OF AIR-BLOWN ASPHALTS AT 25 C. (77 F.).

Asphalt M		Asphalt N		Asphalt O	
Time of Aging, hr.	"Apparent Viscosity," poises × 10^{-6}	Time of Aging, hr.	"Apparent Viscosity," poises × 10^{-6}	Time of Aging, hr.	"Apparent Viscosity," poises × 10^{-6}
2.5	6.44	4.25	3.16	3.25	2.01
24.5	7.30	29.0	3.49	28.0	2.83
49.0	7.51	53.0	3.71	49.0	2.97
120.0	8.31	124.0	3.92	100.0	3.37
336.0	8.87	316.0	4.51	339.0	4.45
698.0	9.61	699.0	5.10	699.0	5.50
1538.0	10.7	1727.0	6.09	1541.0	6.91
3025.0	12.2	3002.0	6.95	3026.0	7.88

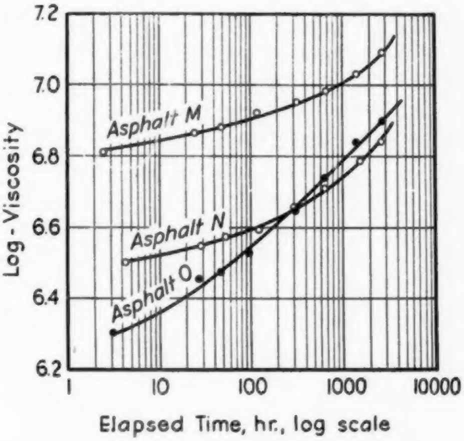

FIG. 1.—Increase of Consistency with Time.

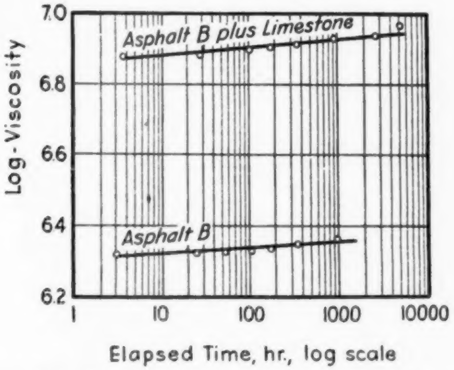

FIG. 2.—Viscosity-Time Relationship for Mixture I.

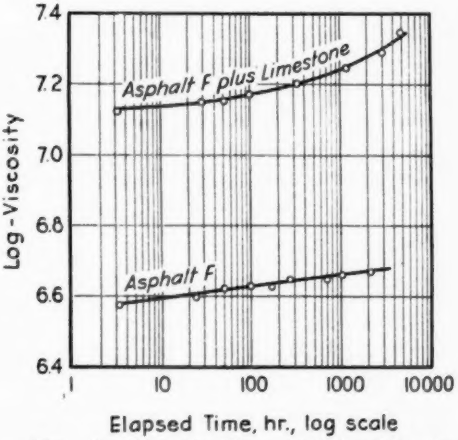

FIG. 3.—Viscosity-Time Relationship for Mixture III.

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[10] A non-Newtonian liquid is one in which the rate of shear is not proportional to the shearing stress.

ent viscosity" would be more nearly comparable. The increases in consistency which occurred with time are indicated in Table III and Fig. 1, where the logarithm of the "apparent viscosity" is plotted against the logarithm of the time of aging.

It is evident that all three of these air-blown asphalts increased in "apparent viscosity" at a much faster rate than those bitumens to which Eq. 1 applies. Figure 1 also indicates that the more highly blown an asphalt, the more rapid is the increase of its consistency with time. The rate of age hardening may be considered as a sensitive measure of differences in degree of internal structure.

TABLE IV.—CHARACTERISTICS OF ASPHALT-LIMESTONE MIXTURES.

50 per cent CS_2-insoluble.

Asphalt				Mixture				
Designation	Source	Process	Penetration, 77 F., 100-g. weight, 5 sec.	Designation	Penetrations			Softening Point, Ring-and-Ball Method, deg. Fahr.
					32 F., 200-g. weight, 60 sec.	77 F., 100-g. weight, 5 sec.	115 F., 50-g. weight, 5 sec.	
B....	Californian	Vacuum	55	I....	6	27	201	127
G....	Fluxed Refined Trinidad Native Lake	....	57	II....	10	33	160	137
F....	Venezuelan	Vacuum	57	III....	8	29	....	136
L....	Venezuelan	Batch steam	53	IV....	14	30	128	139

The increase in consistency of these air-blown asphalts could be partially or wholly eliminated by heating to a temperature above the softening point. It has also been shown elsewhere[11] that continued mechanical working is as effective as heat in eliminating the increased consistency due to age hardening.

ASPHALT-LIMESTONE DUST MIXTURES

Several mixtures (containing 50 per cent CS_2-insoluble matter) composed of asphalt and limestone dust have been studied using the technique employed with the asphalts. The results indicate that the rates at which the consistencies of the mixtures increased with time are chiefly dependent on the nature of the asphalt present. The properties of the asphalts and mixtures are given in Table IV.

The consistencies obtained on these mixtures over a period of several thousand hours are reported in Table V.

Figure 2 is a plot of the logarithm of viscosity against logarithm of elapsed time for mixture I made from Californian asphalt. For comparison, the age-hardening curve for the asphalt is plotted in the same figure. It is apparent that the addition of limestone dust had no appreciable effect on the aging characteristics of this asphalt, since the slopes of the two lines are approximately the same. The limestone-filled Trinidad Native Lake Asphalt Cement (mixture II) was also found to have about the same A.A.I. as the unfilled cement. Mixtures I and II were both essentially viscous liquids.

Mixture III made from a Venezuelan vacuum-refined asphalt of about 50 penetration showed a higher rate of age hardening than the unfilled asphalt as indicated in Fig. 3. The asphalt possesses only slight anomalous flow characteristics and its rate of age harden-

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[11] C. E. Coombs and R. N. Traxler, "Rheological Properties of Asphalts. IV. Observations Concerning the Anomalous Flow Characteristics of Air-blown Asphalts," *Journal of Applied Physics*, Vol. 8, p. 291 (1937).

ing follows the relationship indicated by Eq. 1. However, the mixture showed definite anomalous flow properties and the log-viscosity *versus* log-time plot, like those for air-blown asphalts, was curvilinear.

The Venezuelan oil asphalt refined to 50 penetration in a batch steam still gave a curve on the log-log age-hardening plot (see footnote of Table I). Mixture IV prepared from this asphalt also gave a curve. This mixture possessed distinct anomalous flow characteristics as did the asphalt from which it was prepared.

The formation of structure which results in deviations from viscous flow evidently causes a marked increase in the rate of age hardening. This is true of asphalts and asphalt-mineral mixtures.

STRUCTURE IN ASPHALTS AS INDICATED BY ETCHED SURFACES

It has been claimed[12] that the patterns obtained by the etching of asphalt surfaces for several seconds with solvents could be used to identify the asphalts. Visual evidence of structure in asphalts possessing anomalous flow characteristics and showing rapid age hardening would offer confirmation of the explanations given for these complex flow properties.

In some preliminary work a number of different asphalts have been treated with ethyl ether and 86° Baumé naphtha for a few seconds, the solvent poured off and photographs (at magnification of 220) taken of the dry surfaces using a microscope equipped with an "Ultrapak" attachment. Before etching, the asphalt surfaces were left in contact with air but protected from dust.

Figure 4 shows photomicrographs of surfaces of asphalt L (Venezuelan batch-steam-refined asphalt) which were treated for 5 sec. with ethyl ether. A highly air-blown asphalt of 40 penetration produced from Venezuelan petroleum was treated in the same manner. The etched surfaces 1 hr. and 1 month old are shown in Fig. 5. The character of the structure appears to become more definite with age. It is also apparent that there is a difference in structure between steam-refined and air-blown asphalts. The patterns obtained were not due to surface oxidation because samples sealed to prevent access of air gave similar designs when treated with solvent.

TABLE V.—DATA FOR AGE-HARDENING OF ASPHALT-LIMESTONE MIXTURES AT 25 C. (77 F.).

Mixture I		Mixture II		Mixture III		Mixture IV	
Time of Aging, hr.	Apparent Viscosity, poises $\times 10^{-6}$	Time of Aging, hr.	Apparent Viscosity, poises $\times 10^{-6}$	Time of Aging, hr.	Apparent Viscosity, poises $\times 10^{-6}$	Time of Aging, hr.	Apparent Viscosity, poises $\times 10^{-6}$
3.8	7.58	4.8	8.19	3.5	13.2	3.3	18.2
27.0	7.61	29.5	8.87	29.5	14.0	24.8	20.8
96.8	7.89	51.5	8.93	52.5	14.2	50.5	24.6
168.0	8.01	98.3	9.05	100.0	14.8	98.7	24.5
337.0	8.14	339.0	9.60	340.0	16.0	341.0	26.2
940.0	8.43	1013.0	9.51	1204.0	17.5	2716.0	37.4
2523.0	8.60	2043.0	10.4	3029.0	19.4	4801.0	42.5
				4803.0	22.2	5238.0	47.1

Essentially viscous oil asphalts possessing no marked anomalous flow characteristics and low rates of age hardening, such as asphalts A, B, D and F, gave no evidence of structure when treated in the same manner as the asphalts shown in Figs. 4 and 5.

DISCUSSION

The fact that the rate of increase of consistency with time becomes greater with the degree of anomalous flow exhibited by an asphalt leads to the conclusion that age hardening is directly dependent on the gradual formation of some kind of internal structure. This structure formed with the passage of

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[12] Frank Schwarz, "Etching Experiments upon Asphalts," *Asphalt und Strassenbau*, No. 5, Supplement of *Petroleum Zeitschrift*, Vol. 28 (1932).

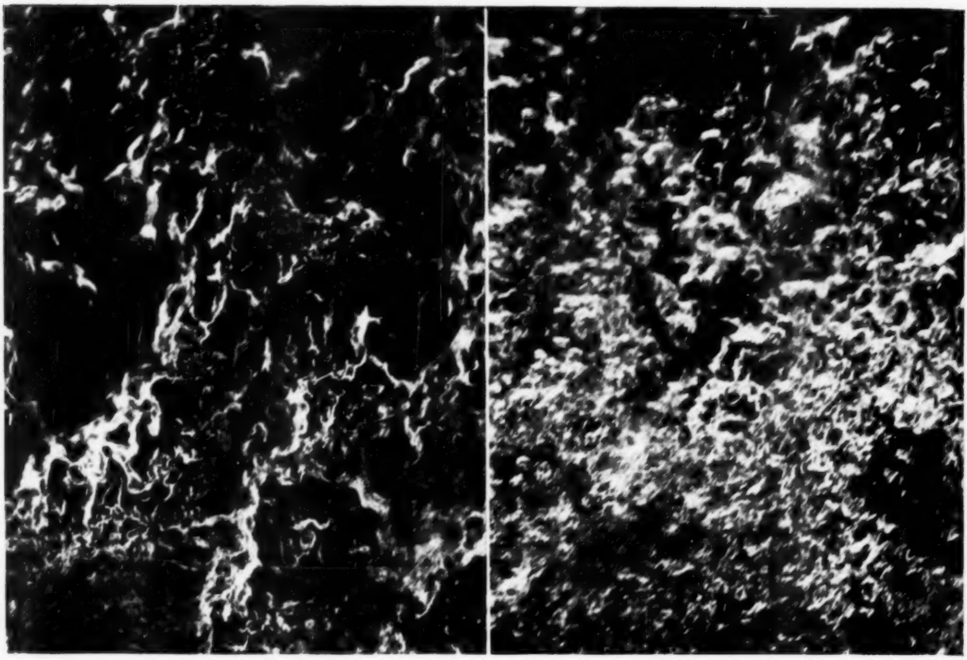
(*a*) Etched 1 hr. after pouring. (*b*) Etched 1 month after pouring.

FIG. 4.—Surfaces of Steam-Refined Asphalt Etched with Ethyl Ether (× 220).

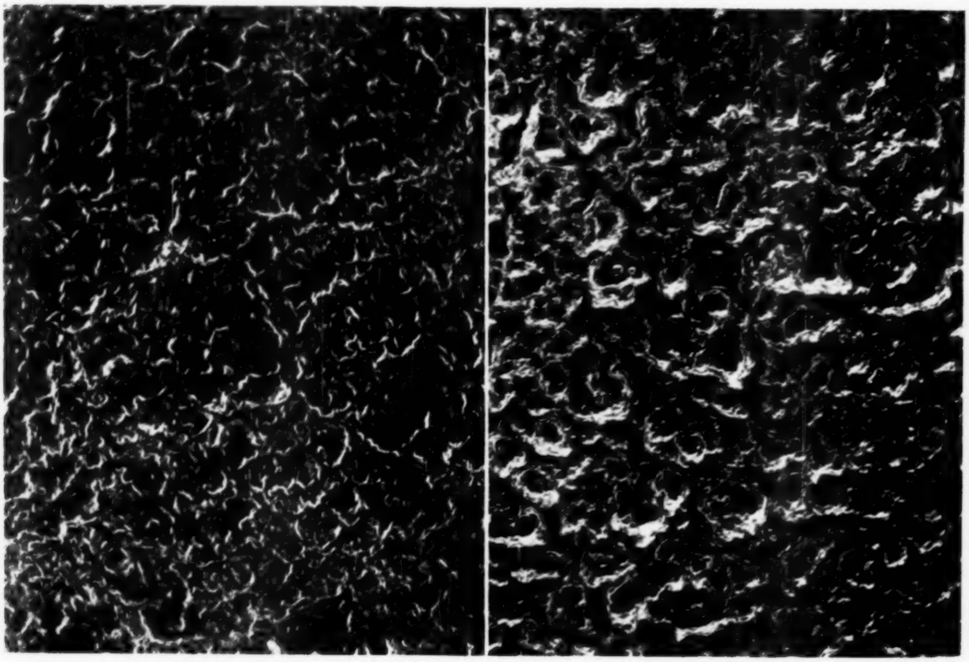
(*a*) Etched 1 hr. after pouring. (*b*) Etched 1 month after pouring.

FIG. 5.—Surfaces of Air-Blown Asphalt Etched with Ethyl Ether (× 220).

time is unstable to heat and mechanical working. Due to its reversible nature, the age-hardening phenomenon may be considered as a form of thixotropy which, together with anomalous flow properties, is typical of many colloidal systems.

The concept of asphalt as a two-phase system with asphaltenes dispersed in the lighter petrolenes as the continuous phase is almost universally accepted and serves well as a basis for the explanation of the formation of internal structure with time. As the temperature is decreased, the asphaltenes become more insoluble in the oily petrolenes. Also, there is apparently a gradual isothermal sol-gel transformation occurring in an asphalt with the passage of time; the internal structure develops rapidly at first and then at a steadily decreasing rate.

Acknowledgment.—The authors are indebted to H. E. Schweyer for the drawings and to C. U. Pittman for valuable assistance in preparing the manuscript.

DISCUSSION

Mr. G. L. Oliensis.[1]—Perhaps it would be well to explain just how the surface is etched for the test.

Mr. R. N. Traxler.[2]—The technique of the test is very simple; the most difficult part is the obtaining of good photographs. We filled lids of 3-oz. penetration tins with the asphalt in question. After it had cooled, a small quantity of the solvent was poured on the surface of the bitumen and left there for 4 or 5 sec. The liquid (containing dissolved material) was then poured or thrown from the surface, and the traces of the solvent which remained permitted to evaporate spontaneously. The sample was then placed under the microscope and examined for the presence or absence of a surface pattern. If no pattern was obtained we gave the asphalt surface another washing with the solvent. Sometimes it required three treatments to get a good pattern. With asphalts that were essentially viscous (for example, California and Trinidad oil asphalts) neither repeated treatment of the surface or one brief application gave a pattern. This was also true if the surface had been allowed to age for several weeks.

We found it necessary to use a solvent that evaporated quickly. Turpentine, para-cymene and a number of other liquids were tried but they did not evaporate rapidly with the result that the asphalt surface was in a soft condition for a long time after the excess solvent had been poured off. Further, we worked on the principle that we needed a solvent which would dissolve the oily portion of the asphalt (the petrolenes) leaving the asphaltenes in a flocculated condition.

Mr. S. R. Church.[3]—Referring to the asphalt-limestone mixtures, was the percentage of limestone added the same in each case?

Mr. Traxler.—No. We studied four different mixtures, all of which were made up to 50 per cent carbon disulfide soluble. One of the mixtures contained Trinidad lake asphalt cement to which less than 50 per cent limestone was added because of the mineral matter naturally present in that asphalt.

Mr. P. M. Van Doormaal.[4]—I should like to ask Mr. Traxler whether he has also found indications of increasing viscosity under higher stresses, for instance, under the shearing stresses prevailing in the penetration test. If the penetration of the asphalts which have been submitted to age hardening is determined, is decrease in penetration found or is the increase in consistency exclusively found under the comparatively low shearing stresses of the tests described in the paper?

Mr. Traxler.—In recent weeks we have used the method of successive penetrations, proposed by Thelen[5] and modified by Rhodes and Volkmann[6], for the study of the age-hardening phenom-

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[2] Research Division, Technical Bureau, The Barber Co., Inc., Maurer, N. J.

[3] Consulting Chemist, New York City.

[4] Chemical Engineer, Shell Development Co., Emeryville, Calif.

[5] E. Thelen, "A Method for Measuring the Rheological Properties of Materials of Great Consistency Such as Asphalts," *Journal of Applied Physics*, Vol. 8, pp. 135–137 (1937).

[6] E. O. Rhodes and E. W. Volkmann, "The Use of the A.S.T.M. Penetration Equipment for Estimating the Viscosities of Materials of High Consistency," *Journal of Applied Physics*, Vol. 8, pp. 492–495 (1937).

enon. We allowed samples to time-harden in penetration tins at constant temperature and then tested by this new method using an A.S.T.M. standard penetrometer. Apparently this method will be a fairly satisfactory means of evaluating the rate of time-hardening. Although the shearing stresses used in the penetrometer method are 10 to 100 times greater than those employed in the falling coaxial cylinder viscometer, the increased consistency due to aging can be detected by this application of the penetrometer.

I should point out that when we investigated non-viscous asphalts in the viscometer we applied the same shearing stress to all samples.

THE CONSTITUTION OF CRACKED AND UNCRACKED ASPHALTS

By E. S. Hillman[1] and B. Barnett[1]

Synopsis

This paper supplies chemical evidence supporting the conclusion drawn from the rheological properties of cracked asphalts, that their greater degree of dispersion as compared with straight-run asphalt can be accounted for by the greater aromaticity of the maltenes. It is further shown that the components in low-level cracked asphalt have in all probability average molecular weights lower than those of the corresponding components in a straight-run residue. The chemical structure of asphaltenes is discussed, together with the probability that they consist of a combination of units comprising cyclic groups of no more than two or three rings condensed together.

Introduction

The work from which the following is taken was primarily intended as a study of the stability of cracked fuels. It has, however, provided indirectly some interesting information on the structure of asphalts, and this information is considered here.

The work of Pfeiffer and van Doormaal[2] and of Mack[3] and others has shown that the properties which most affect the practical applications of asphalts are temperature susceptibility, plasticity, elasticity, etc. These properties constitute the rheological behavior of asphalts. The same authors have shown further that low susceptibility to changes in temperature and a high degree of plasticity and of elasticity are usually found together, the result of incomplete peptization of the asphaltenes, whereas well peptized asphalts are, on the other hand, generally characterized by a high susceptibility with less plasticity and elasticity. At the same time it was observed that although asphalts prepared from cracked residues have considerably higher asphaltene contents than straight-run bitumens of the same penetration from the same crude, the cracked asphalts have higher temperature susceptibilities. Consequently, the cracked asphaltenes must be in a more highly dispersed state than the asphaltenes in straight-run bitumens despite their greater concentration. Since it was known that the dispersing power of the maltenes increases with their aromaticity, it was concluded that the cracked maltenes were probably more aromatic than the straight-run material. Although the evidence was indirect, the conclusion was nevertheless in accord with the general belief that the aromatic content of oils increases in the process of cracking.

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[1] Research Chemist, Shell Development Co., Emeryville, Calif.

[2] J. P. Pfeiffer and P. M. van Doormaal, "The Rheological Properties of Asphaltic Bitumens," *Journal*, Inst. Petroleum Technologists, Vol. 22, No. 152, June, 1936, pp. 414–440; *Kolloid-Zeitschrift*, Band 76, Heft 1, pp. 95–111 (1936).

[3] C. Mack, *Proceedings*, Assn. Asphalt Paving Technologists, Technical Sessions held at Washington, December 9, 1933, p. 40.

TABLE I.—ANALYSES OF DUBBS RESIDUE FRACTIONS.

Viscosity Saybolt Furol 210 F. = 27.8 sec.

Fraction		Internal Pressure of Solvent, atmos. per sq. cm.	Percentage of Original Residue	Chemical Analyses, per cent							Density^{20} C.	Average Molecular Weight
				Carbon	Hydrogen	Nitrogen	Sulfur	Ash	Oxygen	Carbon-Hydrogen Ratio		
Isopentane soluble	Cut No. 10	....	34.17	87.95	10.60	0.3(5)	0.7(0)	Trace	0.40	8.3	0.9681	270^{d}
	Cut No. 9	1180	11.88	87.90	10.00	0.5(5)	0.8(1)	Trace	0.74	8.8	0.9975	345^{d}
	Cut No. 8	1200	4.06	88.65	9.85	0.7(4)	0.8(5)	Trace	0.00	9.0	1.0136	344^{d}
	Cut No. 7	1250	7.09	88.20	9.70	0.7(7)	0.8(8)	Trace	0.45	9.1	1.0220	350^{d}
	Cut No. 6	1330	4.39	88.35	9.40	0.9(7)	0.9(1)	Trace	0.37	9.4	1.0403	353
	Cut No. 5^{a}	1420	7.36	88.45	9.20	0.9(8)	0.8(3)	0.00(8)	0.53	9.6	1.0453	374^{d}
	Cut No. 4^{a}	1520	4.15	88.35	8.85	1.0(2)	0.7(4)	0.01(3)	1.03	10.0	1.0583	384^{d}
	Cut No. 3^{a}	1640	5.76	88.50	8.75	1.1(1)	0.8(3)	0.01(6)	0.79	10.1	1.0683	395^{d}
	Cut No. 2^{a}	1800	3.62	88.25	8.40	1.2(4)	0.9(5)	0.06	1.10	10.5	1.0860	439^{d}
	Cut No. 1^{a}	1900	2.52	88.05	8.10	1.3(7)	0.9(8)	0.30	1.20	10.9	1.1102	483^{d}
Isopentane insoluble	Hexane-soluble^{b}	....	4.2	88.1	7.4	1.8	1.3(3)	0.07	1.30	11.9	1.1–1.2	490^{e}
	CCl$_4$-soluble^{c}	...	6.3	83.9	6.2	2.2	0.3(7)	0.10	6.73	13.5	1.18	830^{e}–870^{f}
	Benzene-soluble	....	2.0	86.8	6.1	2.3	0.8(8)	0.17	3.75	14.2	1.19	880^{f}
	CS$_2$-soluble^{e}	....	1.0	87.1	6.0	2.1	0.9(0)	0.25	3.68	14.5	....	817^{f}
	Pyridine-soluble^{c}	....	2.0	83.7	5.6	3.2	0.9(5)	0.22	6.31	15.1	1.24	860^{f}

[a] Semi-solids at room temperature.

[b] Solid at room temperature. Melts 90 to 100 C.

[c] Powdery solids; the CCl$_4$ solubles melt at high temperatures with decomposition and swelling; the benzene-solubles also decompose, although with less swelling; and the carbenes and carboids neither melt nor swell.

[d] In benzene, at the boiling point and freezing point.

[e] In benzene, at the boiling point; and in naphthalene, at the freezing point.

[f] In naphthalene, at the freezing point.

TABLE II.—ANALYSES OF STRAIGHT RUN RESIDUE FRACTIONS.

Viscosity Saybolt Furol 210 F. = 23.2 sec.

Fraction		Internal Pressure of Solvent, atmos. per sq. cm.	Percentage of Original Residue	Chemical Analyses, per cent							Density^{20} C.	Average Molecular Weight
				Carbon	Hydrogen	Nitrogen	Sulfur	Ash	Oxygen	Carbon-Hydrogen Ratio		
Isopentane soluble	Cut No. 19	....	9.51	86.6	12.7	0.10	0.49	0.01	0.10	6.83	0.9075	293^{b}
	Cut No. 18	800	4.28	86.8	12.4	0.18	0.56	0.02	0.04	7.0	0.9292	373^{b}
	Cut No. 17	845	0.48	86.7	12.4	0.18	0.69	0.02	0.01	7.0	0.9393	365^{b}
	Cut No. 16	855	6.37	86.6	12.3	0.16	0.67	<0.01	0.26	7.05	0.9340	391^{b}
	Cut No. 15	915	1.71	86.3	12.0	0.28	0.56	0.009	0.85	7.19	0.9440	421^{b}
	Cut No. 14	935	4.19	86.7	12.0	0.24	0.70	<0.01	0.35	7.23	0.9460	413^{b}
	Cut No. 13	975	3.04	86.9	12.0	0.34	0.75	0.02	....	7.22	0.9493	427^{b}
	Cut No. 12	1010	3.61	86.4	11.8	0.32	0.81	0.03	0.64	7.3	0.9529	438^{b}
	Cut No. 11	1035	2.19	86.4	11.7	0.36	0.79	0.01	0.74	7.36	0.9593	448^{b}
	Cut No. 10	1055	5.80	86.3	11.5	0.43	0.66	0.02	1.01	7.51	0.9613	448^{b}
	Cut No. 9	1090	5.80	86.5	11.4	0.53	0.79	0.01	0.77	7.58	0.9690	497^{b}
	Cut No. 8	1130	6.84	86.8	11.3	0.56	0.92	<0.01	0.41	7.68	0.9790	489^{b}
	Cut No. 7	1195	3.61	86.8	11.0	0.77	1.15	<0.01	0.27	7.9	0.9934	397^{b}
	Cut No. 6	1235	4.56	86.6	10.8	0.87	0.97	0.05	0.71	8.02	1.0031	520^{b}
	Cut No. 5	1290	2.18	86.8	10.5	0.99	0.97	0.04	0.70	8.26	1.0157	539^{b}
	Cut No. 4	1330	1.90	86.6	10.3	1.08	0.87	0.05	1.10	8.41	1.0234	524^{b}
	Cut No. 3	1360	1.33	86.8	10.1	1.21	0.84	0.20	0.85	8.6	1.0370	541^{b}
	Cut No. 2	1385	1.80	86.6	9.9	1.23	0.95	0.13	1.19	8.76	1.0415	576^{b}
	Cut No. 1	1420	15.80	86.7	9.7	1.29	0.95	0.03	1.33	8.94	1.0479	616^{b}
Hexane-solubles		....	5.6	86.25	8.55	2.03	1.15	0.07	1.95	10.1	1.103	1630^{a}; 850^{c}
CCl$_4$-solubles		....	9.4	84.4	7.6	2.24	1.33	0.97	3.46	11.1	1.116	2400^{b}; 1660^{c}

[a] In benzene, at the boiling point.

[b] In benzene, at the freezing point.

[c] In naphthalene, at the freezing point.

There were, however, difficulties in the way of accepting the above point of view. Evidence against this theory is the instability or apparently poor dispersion of the asphaltenes in cracked fuels, which manifests itself in the well-known sludge deposits that such fuels were once so prone to produce.

However, this poor dispersion is not an unavoidable property of the system. One of the authors had found in 1926 that a characteristic of the high-level residues produced at that time, with their high content of poorly peptized material, was the low proportion of maltene resins and that by extracting them from a large quantity of the residue and mixing back with proportionately less residue, cracked asphalts were obtainable of superior ductility, gloss, etc., indicating a high degree of dispersion. Further, attempts to recover the components from such resin-enriched mixtures by extracting with solvents failed; the carbenes and carboids, always present in high-level residues, could no longer be detected, by the usual methods then existing. In other words, the mutual solubility or dispersibility of the components was such that normal extraction methods failed to separate them, and a separation could be effected only by an elaborate extraction with a carefully graded series of solvents. This was definite evidence that the material comprising what is usually considered the heaviest and most intractable components of the cracked residue could be adequately dispersed.

NATURE OF THE MALTENES

From the observation given in the previous section it was evident that incorrect results would be obtained if the proper solvents were not used for the separation of the components. For this purpose a series of solvents of increasing internal pressure was chosen as follows, in the order shown:

Isopentane
Hexane
Carbon tetrachloride
Benzene
Carbon bisulfide

To these was added pyridine which, owing to its polar nature, is a better solvent for many compounds than even carbon bisulfide.

Theoretically the series should start with ethane or propane, but it was found more convenient to make a rough separation with isopentane; and then, when desired, to divide the isopentane-soluble part into fractions by dissolving in propane and successively adding increments of ethane or methane, removing the precipitate after each addition.

Ultimate analyses and also average molecular weights of fractions obtained in this manner are given in Tables I and II.

In Table I the fractions were obtained from a Dubbs low-level residue from a Californian asphalt base crude. With the exception of the values for the average molecular weights, of which the plot flattens out or may even attain a maximum in the direction of the carboids, it will be seen that there is a regular gradation in the data given, showing the reality of the fractionation achieved. This will be dealt with later.

These results should be compared with those given in Table II for fractions obtained from a straight-run residue of approximately the same viscosity from the same crude. Below the dotted line in each table is the material that would be present in a 65 penetration asphalt for both the cracked and the uncracked residue.

The internal pressure of the solvent

corresponding to the precipitate produced gives a basis for comparison of the fractions.

The greater aromaticity of the cracked maltenes is apparent from the fact that the specific gravity is higher for the same molecular weights, and that precipitates of the same molecular weight are obtained at appreciably higher internal pressure of the solvent, aromatics being characterized by higher internal pressures than paraffins.[4] The determination of the average molecular weights for the maltenes presents no difficulties and they may be taken at their face value.

In Tables III and IV the Waterman[5] analyses are given for those cuts which were not too dark. Here again the greater aromaticity of the cracked fractions is apparent. It should be noted that owing to the dark color only a limited number of the cracked fractions could be examined. However in the maltenes of the 65 penetration cracked bitumen, the first fractions have already reached approximately the same aromaticity as the last fraction of the straight run.

It will be seen that the assumption based upon rheological characteristics and a high degree of dispersion, that cracked residues possess a more aromatic medium than straight run, is well supported by the above observations.

MOLECULAR WEIGHT DATA

The average molecular weights for cracked fractions given in Table I show the anomaly of becoming approximately constant while the carbon-hydrogen ratio and the density continue to increase.

TABLE III.—WATERMAN ANALYSES OF FRACTIONS OF DUBBS ISOPENTANE SOLUBLES.

Fraction	Refractive Index, n^{20}_D	Density, d^{20}	Specific Refraction, r^{20}_D.	Molecular Weight	Aniline Point, deg. Cent.	Per cent by Weight		
						Aromatics	Naphthenes	Paraffins
10	1.549(5)	0.9681	0.3289	270	41.0	36	16	48
9	1.573(2)	0.9975	0.3303	345	42.1	45	8	47
8	1.58 (4)	1.0136	0.3318	344	40.5	47	1	52
7	1.58 (9)	1.0220	0.3297	350	32.5	51	2	47

TABLE IV.—WATERMAN ANALYSES OF FRACTIONS OF STRAIGHT RUN ISOPENTANE SOLUBLE.

Fraction	Refractive Index, n^{20}_D	Density, d^{20}	Specific Refraction, r^{20}_D	Molecular Weight	Aniline Point, deg. Cent.	Per cent by Weight		
						Aromatics	Naphthenes	Paraffins
19	1.5008	0.9075	0.3245	293	71.2	14	35	51
18	1.5136	0.9292	0.3238	373	77.6	18	26	55
17	1.5167	0.9343	0.3237	365	80.1	14	36	50
16	1.5170	0.9347	0.3237	391	77.8	18	32	50
15	1.5221	0.9452	0.3227	421	77.0	20	34	46
14	1.5231	0.9433	0.3239	413	77.8	20	30	50
13	1.5271	0.9493	0.3239	427	77.0	22	29	49
12	1.5301	0.9529	0.3242	438	75.4	24	28	48
11	1.5342	0.9593	0.3241	448	73.0	26	27	47
10	1.5362	0.9613	0.3245	448	72.2	27	24	49
9	1.5422	0.9690	0.3249	497	69.3	32	24	44
8	1.5481	0.9790	0.3245	489	64.9	34	25	41
7	1.5597	0.9934	0.3254	497	62.4	37	22	41
6	1.5693	1.0031	0.3268	520	59.3[a]	42	13	45
5	1.5783	1.0157	0.3269	539	56.6[a]	45	10	45
4	1.5862	1.0234	0.3280	524	55.3[a]	47	6	47
3	1.5946	1.0340[b]	0.3284	541	54.2[a]	48	5	47
2	1.6008[a]	1.0415	0.3288	576	53.4[a]	50	4	46
1	1.6060[a]	1.0479	0.3291	616	52.4[a]	53	1	46

[a] Extrapolated. [b] Interpolated.

It is difficult to say with what reliance the determined average molecular weights of such ill-defined mixtures as the isopentane-insoluble fractions can be accepted as absolute, for the solutions of these fractions in all probability depart appreciably from the ideal solution laws. However, they are considered to have a relative value, for a reproducibility within some 7 per cent was readily obtained on any one sample, even after the lapse of several weeks. Also the determinations were made at several concentrations and the results rejected if the slope of the plot of appar-

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[4] Hildebrand, "Solubility," Second Edition, Chapter 10, Reinhold Publishing Corp., New York City (1936).

[5] J. C. Vlugter, H. I. Waterman and H. A. von Westen, "Improved Methods of Examining Mineral Oils, Especially the High Boiling Components," *Journal*, Inst. Petroleum Technologists, Vol. 21, p. 661 (1935).

ent molecular weight against concentration was too great to allow reliable extrapolation to zero concentration. The values given in the tables are the extrapolated values.

Thus while the absolute values found for the final approximately constant molecular weights must be accepted with caution, the existence of a final constant value is in all probability real.

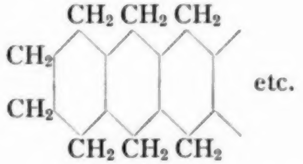
Rather surprisingly the reproducibility of the molecular weights even in different solvents of the cracked fractions was much superior to that of the corresponding straight material. Among the cracked products, values above about 900 were never obtained except with what were obviously unsuitable solvents, that is, solvents for which the slope of the plot of apparent molecular weight against concentration was too great or there was incomplete solution, gelation, etc.

With the straight-run samples, reproducibility on any given sample was good, but in the case of samples with a molecular weight above 1000, anomalies were observed. As in the case of similar determinations on coal hydrocarbons[6] the molecular weights seemed to vary from solvent to solvent, suggesting the possibility of some kind of polymeric aggregation due to a not very strong binding and readily reversible by a sufficiently powerful solvent.

The molecular weight data must be accepted therefore more as an interesting indication and as showing that the asphaltenes in low-level cracked residue for the particular Californian crude examined are of less molecular complexity than the uncracked ones.

THE CHEMICAL SIMILARITY OF CRACKED MALTENES, CRACKED AND UNCRACKED ASPHALTENES

Abraham makes the statement[7] that asphalts have a polycyclic character typified by an extended naphthene structure of the type



Mack[8] speaks of them as polycyclic compounds containing oxygen and sulfur, or both, in bridge or heterocyclic linkage.

Sachanen and Tilicheyev[9] remark that "Due to the fact that the presence of benzene derivatives with long side chains is improbable on account of their instability (to cracking temperatures), increased accumulation of naphthalene derivatives and other condensed aromatic hydrocarbons must be expected." They also point out that the extent to which condensed aromatic hydrocarbons are formed is determined by the depth of cracking.

From the above and similar statements in the literature, it appears to be generally believed that asphaltenes, both cracked and uncracked, are highly aromatic bodies containing more or less condensed aromatic nuclei and that the degree of condensation increases upon cracking.

Now it is known that pure polycyclic compounds such as picene, crackene, and truxene are extremely insoluble in most solvents whereas asphaltenes

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[6] R. C. Smith and H. C. Howard, "Molecular Weights of Polymeric Substances in Catechol and Their Bearing on the Nature of Coal and Derived Products," *Journal*, Am. Chemical Soc., Vol. 58, p. 740 (1936).
R. C. Smith and H. C. Howard, "Equivalent and Molecular Weights of Humic Acids from a Bituminous Coal," *Journal*, Am. Chemical Soc., Vol. 57, p. 512 (1935).

[7] H. Abraham, "Asphalts and Allied Substances," Third Edition, p. 750, D. van Nostrand Co., New York City (1929).

[8] C. Mack, "Colloid Chemistry of Asphalts," Colloid Symposium Monograph, Vol. 10, p. 53, Chemical Catalog Co., New York City (1932).

[9] A. N. Sachanen and M. D. Tilicheyev, "The Chemistry and Technology of Cracking," p. 135, Chemical Catalog Co., New York City (1932).

readily dissolve in benzene, carbon bisulfide, etc. Their solubility in such solvents is not dependent on the presence of resins and the like, as they remain soluble after repeated solution and reprecipitation until the precipitating solvent remains colorless. It can therefore be concluded that in the asphaltenes, cracked or straight, the degree of condensation of the aromatic nuclei is unlikely to be very great although the number of rings present can be large.

In addition to the solubility in benzene, there is the observation that the cracked asphalts, usually assumed to be more highly condensed, possess rheological properties which can only be associated with a lyophilic colloid more highly dispersed than uncracked asphalt systems. These facts throw some doubt upon the conception of a condensed polycyclic structure for asphaltenes, particularly of the type postulated by Abraham.[7]

It has long been held that coal tars can be distinguished from petroleum tars by the fact that the latter resist attack by sulfuric acid. However, if asphaltenes are aromatics, it is surprising that they are unsulfonatable. The explanation appears to be quite simple. They are apparently so strongly protected by adsorbed oily material and resins that the acid cannot penetrate to the asphaltenes in a reasonable time. On the other hand, the highly purified asphaltenes prepared by the extraction method already described are readily sulfonated by fuming acid and rendered completely water-soluble.

Cracked maltenes are also sulfonatable with fuming sulfuric acid, but only part of the straight-run material is attacked. In fact, the original, untreated Dubbs residue used in the experiments could be sulfonated to almost complete solubility in water; only a trace of tarry matter remained undissolved. This indicates that the paraffinic groups shown by the Waterman analyses to be present are probably all attached to aromatic nuclei in the particular sample examined. In other words it appears that the maltene portion of the straight-run residues obtained from this crude oil contains wholly paraffinic molecules as well as paraffin chains attached to aromatic or naphthenic nuclei, whereas in the maltenes from the cracked residues no free paraffins exist, all paraffin groups being present as side chains of aromatic or naphthenic molecules. It would be interesting to investigate how far this is general and whether on cracking waxy crude oils the wax that is left may not be of a ceresinic aromatic character or the only truly paraffinic portions surviving.

Another extremely interesting observation is that all the different fractions from the particular cracked residue examined except the carbenes and carboids give, by sulfonation and liming, calcium salts which qualitatively are identical, having the same appearance, soapy qualities, and extreme solubility in water. Solutions of these salts all exhibit the same curious behavior on evaporation, first becoming syrupy and finally drying rapidly to a powdery consistency.

The above facts suggest that all the constituents of this cracked residue, except possibly the carbenes and carboids, are closely related chemically and are built up of units of some simpler compound or compounds. Even the straight-run asphaltenes with average molecular weights of the order of 2400 in benzene may in the main be referable to such simpler units, and split down to them during sulfonation. It is difficult to conceive of a sulfonated molecule of around 2400 molecular weight giving the thin, clear, non-gel-like solutions in water that are obtained.

A TENTATIVE THEORY OF THE STRUCTURE OF THE CONSTITUENTS OF RESIDUES

The theory to be described is designed primarily as a contribution to the knowledge of the structure of asphalts and is presented not as a necessarily correct theory but only as one formulated during the normal process of development and subject to further investigation. Any theory of the structure of the constituents of bitumen must take into account what is known of the nature of their nearest relatives, the heaviest lubricating oils. The National Bureau of Standards has concluded[10] that in passing from gasoline to oil the paraffin content decreases while the proportions of aromatics and naphthenes increase. The heaviest lubricating oils they examined were considered to comprise chiefly naphthenic molecules with one of more single, double, or triple, etc., naphthenic ring nuclei, with or without paraffin side chains, and aromatic molecules with one or more benzene, naphthalene, anthracene, etc., nuclei, also with or without paraffin side chains.

It is generally accepted that the least soluble cracked fractions do not possess long side chains, and this has to be borne in mind when considering the carbon-hydrogen ratios. The carbon-hydrogen ratios of these fractions are not high enough to permit the presence of large condensed nuclei corresponding to the molecular weights found. The alternative is units of relatively smaller nuclei strung together. Indeed, it is difficult to conceive of any other structure for the highly aromatic asphaltenes of which the molecular weight is from 700 to 800, while the carbon-hydrogen ratio is only 13.5 instead of, for instance, 24.0 for decacyclene, of which the molecular weight is 450. This is particularly true for the straight-run asphaltenes, whose molecular weights are already about 2400 while the carbon-hydrogen ratio is only 11.1. Because of the similarity of the derivatives, such as bromides and sulfonic acids, of the various fractions, it is highly probable that the units which combine to form the larger molecules are limited to a few types that are very similar.

In the process of mild cracking such materials as resins or asphaltenes, the larger structures are broken down into simpler units. At the same time the remaining side chains may be lost, apparently as methane (for which there is experimental evidence), and sensible intradehydrogenation occurs with the possible liberation of gaseous hydrogen in small amount. As a consequence, some of the cracking products are not only less aliphatic (accounting for the rise in the carbon-hydrogen ratio) but also more condensed. Since the highly condensed aromatics with over four or five benzene rings are known to be only slightly soluble in even the best solvents, the decreased solubility of some fractions after cracking becomes understandable.

On the other hand the possibility, for the bulk of the products, of a simple breakdown very analogous to depolymerization accompanied by only slight condensation, is not excluded. The literature indicates that in general condensation requires more drastic cracking conditions than depolymerization, so that depolymerization could be the first step in the cracking of such materials as resins and asphaltenes. Under mild cracking conditions, therefore, as in the preparation of low-level residue from straight-run residue, depolymerization may be expected to be the preliminary reaction occurring among the bitumens

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[10] F. D. Rossini, "Fundamental Research on the Chemical Constitution of Lubricating Oil—A.P.I. Research Project 6," *Proceedings*, Am. Petroleum Inst., Vol. 16M, (III), p. 63 (1935).

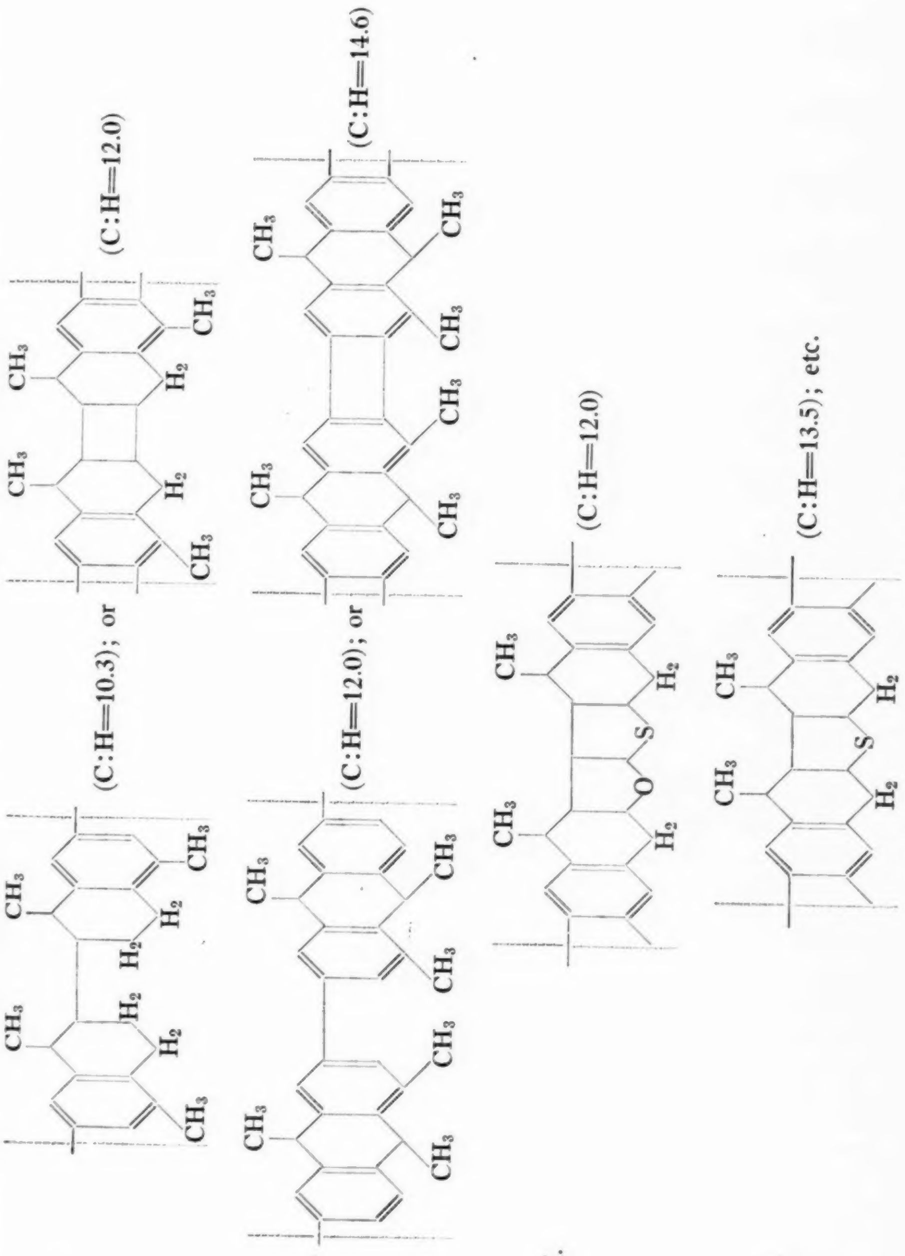
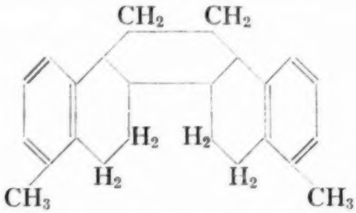


FIG. 1.—Structures of Resins and Asphaltenes.

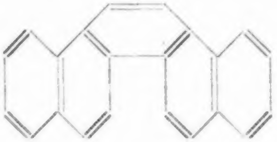
accompanied or almost immediately followed by loss of side chains, and the constituents of such residues should be chemically similar and differ only as members in a homologous series differ in such properties as molecular weight, density, and length of side chains. In fact, the constituents of low-level residue up to and including the asphaltenes actually do behave in this way. The lower solubility of the cracked as compared with the uncracked asphaltenes can be accounted for sufficiently by the decrease in paraffinicity due to loss of side chains. The very small amounts of heavier fractions (carbenes and carboids) present in the low-level residue are to be ascribed to the effect of the condensation reaction which under low-level cracking conditions is apparently incompletely "screened out."

The matter given in the preceding pages, together with much data not conveniently presented here, appear to indicate that resins and asphaltenes may have structures such as shown in Fig. 1. The above are more representative of cracked than straight-run asphaltenes in which there are probably longer side chains corresponding to lower carbon-hydrogen ratios. Although these structures have been represented as identical polymers, the possibility that the units of the resin and asphaltene molecules may be dissimilar is not excluded. Such molecules in which the units are unlike may be called quasi polymers.

A specific application of the theory may not be without interest. Let it be assumed that straight-run CCl_4 solubles comprise polymers, or quasi polymers of the type I. It is not claimed that straight-run asphaltenes actually have this structure; it suffices that the structure assumed is in accord with the known facts. The first result of cracking the asphaltenes is a splitting of the asphaltene polymer into one or more sets of *n*-membered-ring units. A four-membered-ring unit has been indicated above. This splitting may be accompanied by a condensation of the side chains forming:



At the same time the naphthene rings are dehydrogenated with an accompanying shift of hydrogen on losing side-chains:



$+ 3H_2 + 2CH_4$

The final product is picene, molecular weight 278, only slightly soluble in benzene and somewhat more soluble in pyridine. It is improbable that as much hydrogen as is indicated actually escapes as gas.

Perhaps it is not without significance that picene has actually been isolated from a California cracked residue.[11]

While the above may appear to suggest a very simple and perhaps long-chain molecule, we do not pretend that such exists in asphalt. Rheological properties suggest a compact three-dimensional structure in which the above type of unit is probably rolled up upon itself or so connected that some of the units are close to each other in spatial configuration and are more or less united by the residual valences and polar forces in the unsymmetrically substituted rings.

Acknowledgment.—The acknowledgments of the authors are due to the Directors of the Shell Development Company for permission to publish the above work carried out at their laboratories in Emeryville, Calif.

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[11] C. Graebe and J. Walter, "On Picene," *Berichte*, Vol. 14, p. 175 (1881).

DISCUSSION

MR. C. MACK[1] (*presented in written form*).—As far as the writer is aware, the work by Messrs. Hillman and Barnett represents the first attempt to separate asphalts into different cuts by means of fractional precipitation. It is to be regretted that the authors did not include in their paper a detailed description of their technique. The ultimate analysis and mean molecular weights of the different fractions from the Dubbs and straight-run residues confirm the assertion that the aromatic content of oils increases in the process of cracking. Based upon his own unpublished work, the writer agrees with the authors that asphaltenes from a cracked residue have lower molecular weights than those from the corresponding residual crude oil used as cracking stock. It may be stated, however, that the cracking of asphaltene free stocks such as gas oil, produce asphaltenes of higher molecular weight than the heaviest fraction of the stock. The molecular weights of such asphaltenes are still lower than those from cracked reduced crude oil, and have been found to be in the neighborhood of 500.

The authors applied the Waterman analysis to fractions from the cracked as well as straight-run residue. This method is based upon the change in the refractive index brought about by hydrogenation and allows the determination of the percentage of aromatic rings, naphthenic rings and paraffins or paraffinic side chains. The Waterman analysis carried out on fractions 7 to 10 of the cracked residue, with mean molecular weights ranging from 270 to 350, shows paraffinic side chains in the neighborhood of 50 per cent. Since the cracking operation breaks up long paraffinic side chains, it can be expected that the paraffinic groups in the fractions of the cracked material are restricted to methyl groups which the authors apparently also admit in their discussion of the structure of the constituents. In view of the fact that aromatic hydrocarbons are less easily sulfonated the more paraffinic side chains they contain, the results of the Waterman analysis apparently do not agree with the authors' statement that the untreated cracked residue could be sulfonated to almost complete solubility in water. The writer uses with success the determination of the total surface energy of asphalt fractions for obtaining an insight into their chemical structure, and wonders whether the authors have given thought to this method.

The tentative theory of the structure of the constituents of residues outlined in the paper represents a valuable approach to what can be known about the chemical structure of asphaltic constituents.

MR. B. BARNETT.[2]—It is very satisfying that Mr. Mack is in agreement with our conclusion that asphaltenes from a cracked residue have lower molecular weights than those from the

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[1] Research Chemist, Technical and Research Division, Imperial Oil Limited, Sarnia, Ont., Canada.

[2] Research Chemist, Shell Development Co., Emeryville, Calif.

corresponding residual crude oil used as cracking stock.

That the groups attached to the aromatic nuclei are not long is agreed, but we rather suspect that although methyl groups may comprise a large proportion of the side chains, other short groups are also present.

With regard to Mr. Mack's remarks on the apparent discrepancy between the results of the Waterman analyses of the lighter fractions and the possibility of sulfonating the cracked material to complete solubility in water, it must be recalled that the process of sulfonation is always accompanied by extensive oxidation; many of the side chains are undoubtedly oxidized to carboxyl or other oxygen-containing groups, for the final sulfonated product has always been found to contain more oxygen than corresponds to the sulfur present. As a result of this conversion the sulfonation reaction may be hastened. The reaction with straight-run material is slower, probably because of the presence of free paraffins; and for this reason also this material does not become completely water soluble in a reasonable time.

We expect to publish shortly the technique of fractional precipitation used to obtain the several fractions described in our paper.

MR. A. B. HERSBERGER.[3]—I should like to ask whether the apparently abnormally high molecular weights obtained for some of the asphaltenes in benzene solutions can be accounted for by the possibility that they may not be completely soluble in benzene.

MR. BARNETT.—I doubt that they are completely soluble in benzene, but there is no way of proving it and the values given are averages for the conditions of the experiments. That is all we can say about them.

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[3] The Atlantic Refining Co., Philadelphia, Pa.

THE CONSTITUENTS OF ASPHALTIC MATERIALS *versus* ACCELERATED WEATHERING

By R. R. Thurston[1]

Synopsis

Approximately 200 F. melting point asphalts prepared from Mexican, Gulf Coast, and Mid Continent crude and a cracked residue have been separated into asphaltenes, resins and oils.

Synthetic asphalts have been prepared by recombining these constituents in various proportions and from different sources. Accelerated weathering data were obtained on these materials.

Results indicated an increased resistance to weathering when increased proportions of resins were used. Good weathering is dependent not only upon the character and source of the resins and oils but also upon the quality of the asphaltenes. These constituents from different asphalts vary widely in their characteristics.

It is generally known that asphalt can be considered as a mixture of three major groups of constituents, asphaltenes, resins and oils. All asphalts contain varying proportions of these constituents which may be separated from each other by the method of Marcusson.[2] Asphaltenes can be obtained by precipitation from a dilute naphtha solution. The clear solution containing resins and oils is decanted and the asphaltenes may be purified by reprecipitation and obtained as a brown powder by evaporation of the retained naphtha after decantation or filtering. Distillation of this naphtha solution leaves a mixture of resins and oils which is called petrolenes. These are absorbed on fuller's earth, and extraction with naphtha will remove the oils. Subsequent extraction with carbon bisulfide will remove most of the resins. Oils and resins are obtained by evaporation of their respective solvents. Asphalts also contain small quantities of acids which have been neglected in this particular investigation.

Asphaltenes obtained from various sources vary quite widely in molecular weight and other characteristics. They are complex hydrocarbons having a high carbon-hydrogen ratio and probably contain a small amount of oxygen. Resins and oils may be further separated by means of various selective solvents, but for the purposes of this work, resins and oils as obtained above have been used.

Synthetic asphalts were prepared by combining asphaltenes, resins and oils from various sources in varying proportions. This was accomplished by dissolving the three constituents in benzol and removing the benzol by the Abson method.[3] The resulting asphalts have

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[1] Chemical Engineer, Technical Division, The Texas Company, New York City.

[2] J. Marcusson, "Die Naturlichen und kunstlichen Asphalte," p. 92, Verlag von Wilhelm Engelman, Leipzig (1931).

[3] Gene Abson, "Method and Apparatus for the Recovery of Asphalt," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 704 (1933).

been tested for melting point, penetration at 77, 32, and 115 F. and asphaltene content. Exposure panels of these asphalts have been made and tested in an accelerated weathering tester.

Panels used for this work were 3 by 6 in. aluminum, 0.05 in. thick. Asphalts were applied hot to one side of these panels to a thickness between 0.025 and 0.030 in. These were exposed to an Atlas arc lamp at a distance of 18 in. for 18 hr. per day. They also received two freezing periods and two water-spray periods per day. The lamp was operated with and without a water spray on the panels on alternate days. Panels were inspected daily and considered as failed when the metal showed through the asphalt film in more than one spot or crack. The average temperature of the panels during exposure to light without a water spray was 140 F. and for this reason all asphalts prepared were close to 200 F. in melting point.

TABLE I.—ASPHALTS USED.

	STEAM-REDUCED MEXICAN	AIR-BLOWN MEXICAN	AIR-BLOWN GULF COAST	AIR-BLOWN MID-CONTINENT	AIR-BLOWN CRACKED RESIDUE
Melting point, deg. Fahr., ring-and-ball method.......	157	204	201	199	202
Ductility, 77F........	16	5	2	1	....
Penetration, 77 F.....	18	16	18	25	1
32 F.....	9	9	12	13	0
115 F.....	66	36	33	46	4
Asphaltenes, per cent.	30.3	41.6	33.8	28.3	52[a]

[a] Carbene free.

Table I shows physical properties and asphaltene contents of the various original asphalts used in this work. Both steam-reduced and air-blown asphalts prepared from Mexican crude were used. Air-blown asphalts prepared from Gulf Coast and Mid-Continent crudes were used and also an air-blown asphalt from a highly cracked residuum. This latter product contained a considerable proportion of carbenes which were not used in preparing any of the synthetic asphalts included in this investigation.

Table II shows a series of asphalts made up exclusively of the constituents from an air-blown Gulf Coast residuum.

Sample No. 1 is a straight asphalt as produced, and No. 2 is this same asphalt made synthetically by recombining the constituents after separation. This pair of samples serves as a check on the procedure and it is seen that the synthetic product weathered only 80 per cent as long as the original, which would indicate that the procedure of separating and recombining the constituents is not perfect.

Sample No. 3 contains an increased proportion of resins and has weathered over twice as long as sample No. 2 and even 65 per cent longer than the original sample No. 1.

Samples Nos. 4 and 5 contain increased proportions of asphaltenes and weathered distinctly less satisfactorily than sample No. 2.

Sample No. 6 contains an increased proportion of oils and weathered less satisfactorily than sample No. 2 in spite of the fact that this increased amount of oils markedly increased the penetration of the product and reduced the susceptibility.

It can evidently be concluded that increasing the proportion of resins in this particular type of asphalt improves the resistance to weathering and that increasing the proportion of asphaltenes or oils reduces the resistance to weathering.

Table III shows a series of asphalts all made from asphaltenes obtained from Mexican crude oil combined with petrolenes from various sources.

Sample No. 1 is the original air-blown Mexican asphalt and sample No. 2 is the same product after separation of the constituents and recombining. Here again resistance to weathering was reduced by the procedure of synthetically preparing sample No. 2.

Sample No. 3 is made from the asphaltenes from air-blown Mexican asphalt and petrolenes from air-blown cracked residuum. It is seen that the weathering characteristics are seriously injured by the use of these petrolenes.

TABLE II.—AIR-BLOWN ASPHALTS FROM GULF COAST RESIDUUM.

	Sample No. 1—Straight Asphalt	Sample No. 2—Sample No. 1 Separated and Recombined	Sample No. 3—225 F. Melting Point Asphalt Plus 13 per cent Resins from Sample No. 1	Sample No. 4—170 F. Melting Point Asphalt Plus 8 per cent Asphaltenes from Sample No. 1	Sample No. 5—145 F. Melting Point Asphalt Plus 16.6 per cent Asphaltenes from Sample No. 1	Sample No. 6—225 F. Melting Point Asphalt Plus 19 per cent Oils from Sample No. 1
Melting point, deg. Fahr., ring-and-ball method	201	201	201	203	213	200
Penetration, 77 F.	18	18	16	14	10	28
32 F.	12	12	8	7	7	20
115 F.	35	33	27	24	18	46
Asphaltenes, per cent	34.7	34.7	33.2	36.3	38.7	31.2
Resins, per cent	13.3	13.3	23.8	17.4	16.7	10.3
Oils, per cent	50.0	50.0	43.0	46.3	44.6	58.5
Accelerated weathering test, days to failure	161	128	265	91	44	95
Panel number	100	207	98	97	101	99

TABLE III.—ASPHALTENES FROM MEXICAN—PETROLENES FROM VARIOUS SOURCES.

	Sample No. 1	Sample No. 2	Sample No. 3	Sample No. 4	Sample No. 5	Sample No. 6	Sample No. 7	Sample No. 8
Asphaltene source	Air-Blown Mexican	Air-Blown Mexican	Air-Blown Mexican	Steam-Reduced Mexican	Steam-Reduced Mexican	Steam-Reduced Mexican	Steam-Reduced Mexican	Steam-Reduced Mexican
Petrolene source	Air-Blown Mexican	Air-Blown Mexican	Air-Blown Cracked Residue	Steam-Reduced Mexican	Air-Blown Mexican	Air Blown Gulf Coast	Air-Blown Mid-Continent	Air-Blown Cracked Residue
Melting point, deg. Fahr., Ring-and-ball method	204	206	201	197	208	212	250	191
Ductility, 77 F.	5	3	2	1	2	1	2	1
Penetration, 77 F.	16	18	12	7	10	15	27	7
32 F.	9	12	7	3	6	10	18	2
115 F.	36	38	35	38	22	21	35	23
Asphaltenes, per cent	41.6	42.1	49.7	44.4	45.6	39.1	33.7	63.0
Accelerated weathering test, days to failure	318	289	48	34	59	24	48	34
Panel number	194	205	209	200	210	206	216	199

Samples Nos. 4, 5, 6, 7 and 8 are all made from asphaltenes from steam reduced Mexican asphalt and petrolenes from various sources. It is well known that straight steam-reduced Mexican asphalt of this high melting point does not resist this type of weathering test, although soft grades are excellent paving asphalts. This is partially due to extreme hardness and high susceptibility, as indicated by sample No. 4.

Accelerated weathering is somewhat improved when petrolenes from air-blown Mexican are used as in sample

No. 5 but does not approach the results obtained on sample No. 2 made entirely from air-blown Mexican.

Weathering is still inferior when other petrolenes are used, as in samples Nos. 6, 7 and 8.

These data show that the source of petrolenes has a definite bearing on weathering, but the source of the asphaltenes also has a very definite effect.

A series of asphalts was attempted using petrolenes from various sources and asphaltenes from air-blown cracked residuum, but homogeneous products could not be obtained.

TABLE IV.—PETROLENES FROM MEXICAN—ASPHALTENES FROM VARIOUS SOURCES.

	SAMPLE NO. 1	SAMPLE NO. 2	SAMPLE NO. 3
ASPHALTENE SOURCE...	STEAM-REDUCED MEXICAN	AIR-BLOWN GULF COAST	AIR-BLOWN MID-CONTINENT
PETROLENE SOURCE..	STEAM-REDUCED MEXICAN	STEAM-REDUCED MEXICAN	STEAM-REDUCED MEXICAN
Melting point, deg. Fahr., ring-and-ball method......	197	200	199
Ductility, 77 F......	1	3	0
Penetration, 77 F...	7	10	9
32 F...	3	5	6
115 F...	38	23	22
Asphaltenes, percent..............	44.4	43.5	39.6
Accelerated weathering test, days to failure............	34	148	66
Panel number.......	200	208	214

Table IV shows asphalts prepared from asphaltenes from various sources combined with petrolenes from steam-reduced Mexican asphalt.

Sample No. 1, made with constituents entirely from steam-reduced Mexican asphalt in their normal proportions weathers poorly, as shown in Table III.

Sample No. 2, made with asphaltenes from air-blown Gulf Coast asphalt and petrolenes from steam-reduced Mexican weathers distinctly better than sample No. 1, and sample No. 2 made with the asphaltenes from air-blown Mid-Continent residuum and petrolenes from steam-reduced Mexican also weathers better than sample No. 1.

These data indicate that asphalts made with petrolenes from steam-reduced asphalt and asphaltenes from air-blown Mexican asphalt are superior in weathering properties to asphalts made with the same petrolenes and asphaltenes from steam-reduced Mexican asphalt. Asphaltenes produced by oxidation appear to be superior in weathering properties to those originally present in asphaltic crudes.

This work indicates a definite possibility of improving asphalts for some specific purposes where special characteristics are desired. There is a large amount of work to be done along similar lines which may lead to a wider utilization of asphaltic products. Better methods of evaluation are desirable since it is evident that the constituents from no two sources are identical. Asphaltenes are not a definite series of compounds but their characteristics and value vary widely with the source and manner of refining. The quality of resins and oils in different asphalts is also variable and the accelerated weathering test is only an empirical method of evaluation. Adequate scientific explanations for the results reported in this paper are needed, and the necessity for further investigation is indicated.

DISCUSSION

Mr. P. M. Van Doormaal.[1]—I should like to ask whether the picture of the weathering was the same in all cases. Mr. Thurston gave a number of days after which failure occurred. Is the picture of the failure in every case the same?

Mr. R. R. Thurston.[2]—We have defined failure as the number of daily cycles at which the metal shows through the asphalt in at least two spots which are further than $\frac{1}{4}$ in. from the edge of the panel. The patterns at the time of failure are not the same but vary in regard to the number and size of the developed cracks, checking, and general surface appearance.

Mr. V. A. Endersby.[3]—I should like to ask whether any attempt has been made to correlate the pattern of weathering with the different constituents of the material.

Mr. Thurston.—Asphalts from straight crude oil sources consistently show characteristic pattern at failure. There seems to be no correlation, however, of the failure patterns on these synthetic products where we have crossed up so many constituents from various sources.

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[1] Chemical Engineer, Shell Development Co., Emeryville, Calif.

[2] Chemical Engineer, Technical Division, The Texas Company, New York City.

[3] Research Engineer, Shell Development Co., Emeryville, Calif.

THE COMPOSITION OF AMERICAN STEAM-DISTILLED WOOD TURPENTINE AND A METHOD FOR ITS IDENTIFICATION

BY T. C. CHADWICK[1] AND S. PALKIN[1]

American steam-distilled wood turpentine generally falls well within the requirements of the Society's Standard Specifications for Spirits of Turpentine (D 13 - 34).[2] Although the values for its refractive index and specific gravity (see Table I) are generally lower than the corresponding values for gum spirits, these properties do not clearly distinguish them. Their boiling range is not very dissimilar. Its odor has long been regarded as a characteristic and distinguishing feature of steam-distilled wood turpentine, but continued improvement in the process of producing and refining has rendered this distinction less and less obvious. The fact that the distinction is less obvious has possibly led to the belief on the part of some that the composition of the two kinds of turpentine is the same or nearly the same. However, as has been found by recent examinations,[3,4] there is a marked difference in composition between gum spirits and steam-distilled wood turpentine.

Dupont and his coworkers[3] fractionally distilled American steam-distilled wood turpentine and determined the Raman spectra of the fractions. They suggest this procedure as a method for identifying the monocyclic constituents—dipentene, terpinene, and terpinolene—characteristic of this kind of turpentine.

Where such apparatus is available, this method is no doubt useful. The method herein proposed is based on the identification by chemical means of the aldehydic and terpene alcohol constituents, which characterize this kind of turpentine.

In the investigation on the composition of steam-distilled wood turpentine made in this laboratory, 15 kg. of a commercial steam-distilled wood turpentine meeting the specifications of the Society were subjected to a careful fractional distillation under reduced pressure. A systematic examination was made of the physical and chemical properties of the fractions so obtained. The constituents were identified by characteristic derivatives. Detailed descriptions of distillation apparatus, procedures, and the chemical methods used for identification of constituents are being published elsewhere.[4] The methods used for the identification of some of the constituents characteristic of steam-distilled wood turpentine serve as a basis for the chemical testing scheme herein proposed.

In this paper only the summary data and conclusions on the composition[5] of steam-distilled wood turpentine are

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[1] Assistant Chemist, and Senior Chemist, respectively, Naval Stores Research Division, Bureau of Chemistry and Soils, U. S. Department of Agriculture, Washington, D. C.

[2] 1936 Book of A. S. T. M. Standards, Part II, p. 742.

[3] G. Dupont, Mme. Rambaud and M. Bonichon, "Sur la Composition de l'Essence Americaine Bois" ***Bulletin*** de l'institut du pin, Vol. 3, No. 6, p. 121 (1935).

[4] S. Palkin, T. C. Chadwick and M. B. Matlack, "Composition and Fractionation of American Steam-Distilled Wood Turpentine," ***Technical Bulletin No. 596***, U. S. Dept. Agriculture, Washington, D. C., November, 1937.

[5] E. K. Nelson, of the Bureau of Chemistry and Soils, showed the presence of benzaldehyde, fenchyl alcohol, borneol and terpineol in steam distilled wood turpentine, April, 1927, unpublished report.

given. The turpentine was shown to consist of the following:

- Dicyclic hydrocarbons:
  - α-pipene (about 80 per cent of the turpentine)
  - β-pinene and camphene (very small quantities).
- Monocyclic hydrocarbons (constituting most of the remainder of the turpentine) including terpinene, terpinolene, dipentene (limonene).
- Terpene alcohols including fenchyl alcohol, borneol, α-terpineol (present in small quantities).
- Other compounds including low-boiling paraffin hydrocarbons, benzaldehyde, furfural, cineol, sobrerol (present only in traces).

Very small quantities of phenols and phenol ethers are present, of which only methyl chavicol (or anethol) was identified. The presence of traces of camphor and cymene are probable. Small quantities of polymerization and oxidation products are also present.

In gum spirits, however, as was shown in a previous publication,[6] virtually all of the hydrocarbon portion consists of dicyclic terpenes, about two-thirds of which are α- and one-third β-pinene.

The difference in gross composition of the two turpentines, while not evident from the usual routine physical tests, becomes manifest on fractional distillation, as may be seen from Figs. 1 to 6.

Distribution, with respect to boiling range at 20 mm., of fractions of steam-distilled wood turpentine and of gum spirits, is shown in Figs. 1 and 2, respectively. As may be noted, the percentage distilling around 50 to 52 C., the α-pinene region, and the percentages distilling around 57 to 59 C., the β-pinene region, respectively, differ markedly in the two turpentines. The optical rotation curves (Figs. 3 and 4) and the refractive index curves (Figs. 5 and 6) show marked differences in form of those parts after the α-pinene.

Table I.—Properties of Samples of Turpentine by A. S. T. M. Tests.

Sample[a]	Kind of Turpentine[b]	Specific Gravity, 15.5/15.5 C.	Refractive Index, $N_D^{20C.}$	Initial Temperature at 760-mm. Pressure, deg. Cent.	Distilling Below 170 C. at 760-mm. Pressure, per cent	Distilling Below 180 C. at 760-mm. Pressure, per cent
No. 1	S.D.W.	0.863	1.467	154	92	....
No. 2	S.D.W.	0.861	1.466	154	98	....
No. 3	S.D.W.	0.861	1.468	155	92	....
No. 4	S.D.W.	0.861	1.468	155	91	....
No. 5	S.D.W.	0.861	1.467	153	98	....
No. 6	S.D.W.	0.861	1.467	154	91	....
No. 7	S.D.W.	0.863	1.468	155	91	....
No. 8	S.D.W.	0.862	1.467	154	88	....
No. 9[c]	S.D.W.	0.861	1.465	152	83	....
A	Gum	0.869	1.471	157	94	....
B	Gum	0.867	1.473	156	94	....
C	Gum	0.869	1.471	157	93	....
D	Gum	0.868	1.472	156	93	....
E	D.D.	0.860	1.475	148	79	94
F	Sulfate	0.863	1.468	157	90	....
ASTM Specification	Gum; S.D.W., and Sulfate	0.860 to 0.875	1.465 to 1.478	150 to 160	90 min.	....
ASTM Specification	D.D.	0.860 to 0.875	1.463 to 1.483	150 to 157	60 min.	90

[a] Sample numbers represent products from different producers.
[b] S.D.W. = steam-distilled wood turpentine, D.D. = destructively distilled turpentine.
[c] Does not conform with Society's specification for S.D.W.

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[6] S. Palkin, "The Fractionation of American Gum Spirits of Turpentine and Evaluation of Its Pinene Content by Optical Means," *Technical Bulletin No. 276*, U. S. Dept. Agriculture, Washington, D. C., January, 1932.

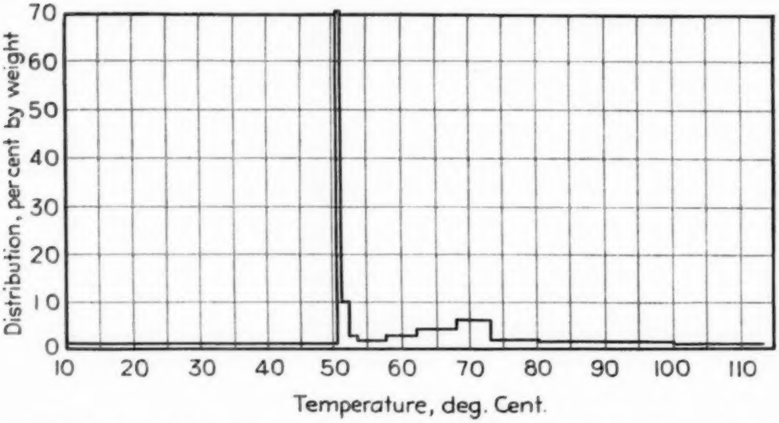

FIG. 1.—Distribution with Respect to Boiling Range at 20 mm. of Fractions of Steam-Distilled Wood Turpentine.

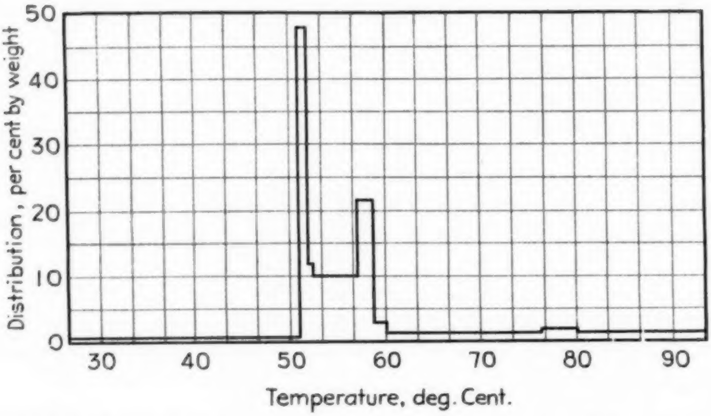

FIG. 2.—Distribution with Respect to Boiling Range at 20 mm. of Fractions of Longleaf Gum Spirits.

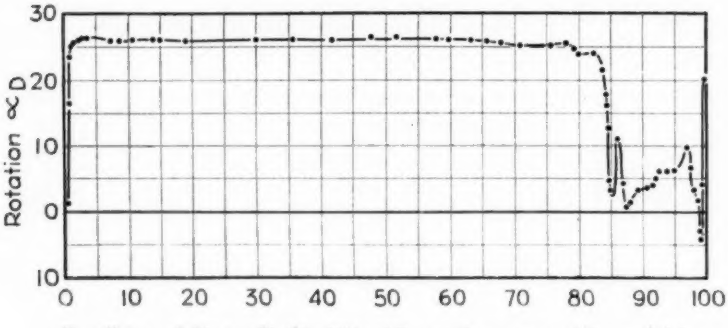

FIG. 3.—Rotation with Respect to Per Cent by Weight, of Fractions of Steam-Distilled Wood Turpentine.

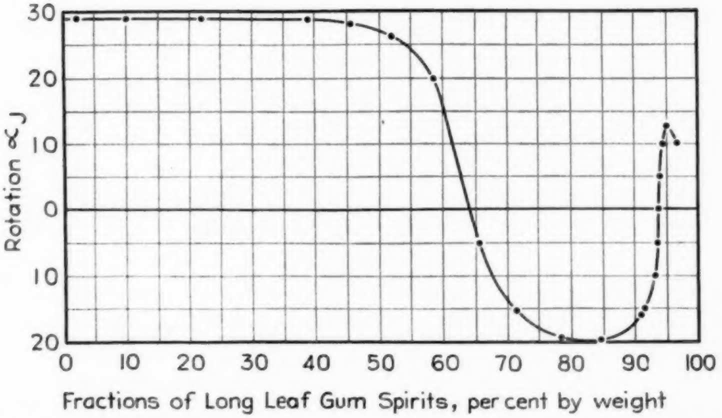

FIG. 4. Rotation with Respect to Per Cent by Weight of Fractions of Longleaf Gum Spirits.

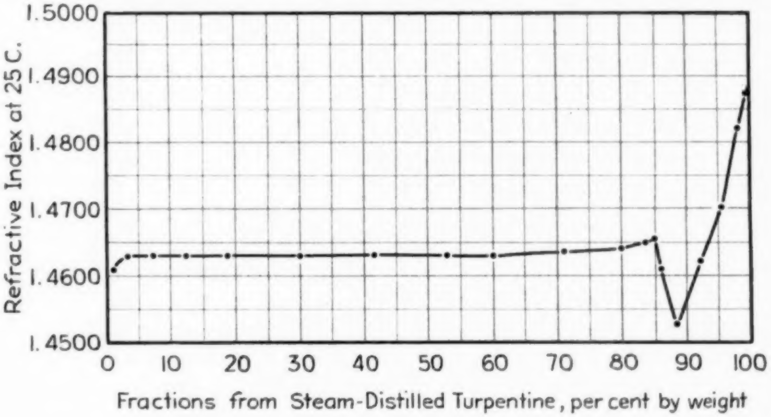

FIG. 5.—Refractive Indices of Fractions from Steam-Distilled Turpentine.

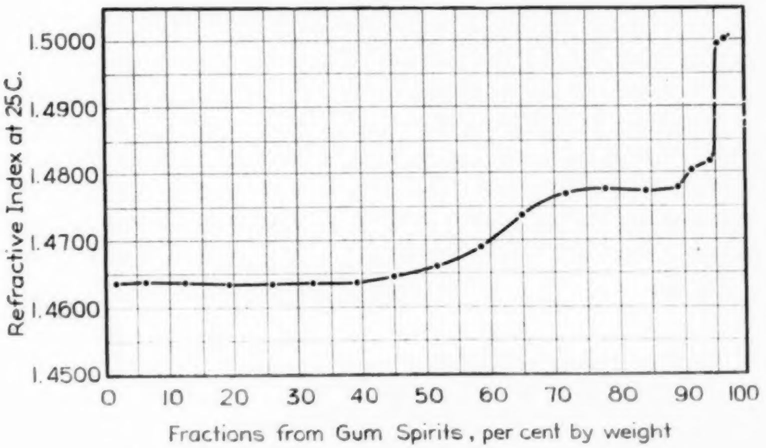

FIG. 6.—Refractive Indices of Fractions from Gum Spirits.

In this connection, it must be remembered that whereas gum spirits represents the whole of the steam volatile oil of pine gum and is normally of reasonable uniformity in composition, turpentines from other sources, such as steam-distilled wood turpentine, are in the nature of fractions, the composition of which is dependent not only on the composition of the oleoresin contained in the resinous wood, which is different from that of the exuded gum oleoresin, but also upon the degree of rectification and refinement to which the turpentine has been subjected.

Detailed procedures are given under tests *a* and *b* described below for establishing the presence or absence of benzaldehyde and fenchyl alcohol, two characteristic constituents present in all samples of steam-distilled wood turpentine meeting the Society's specifications thus far examined.

The proposed tests provide for the use of a 250-ml. sample of turpentine. In some instances 80 ml. were found sufficient. However, the adequacy of the size of sample must be considered in terms of the commercial product, prepared under present-day commercial practice. Any improvement in the rectification and refinement of steam-distilled wood turpentine involving substantial elimination of benzaldehyde and fenchyl alcohol will obviously necessitate the use of larger size samples for the test. The refinement may conceivably even be carried to the point where the quantity of sample required would exceed the laboratory facilities for handling it analytically.

The following is a brief outline of the principles upon which the schematic treatment of the turpentine is based:

A sample of the turpentine is extracted with bisulfite solution to remove aldehydes. The aldehydes, liberated from the bisulfite extract, are steam distilled, and the distillate is extracted with petroleum ether. The petroleum ether extract of the aldehydes is washed with water to separate more completely the benzaldehyde from furfural. The benzaldehyde, after evaporation of solvent, is identified by converting to the 2,4 dinitrophenylhydrazone. The aldehyde-free turpentine is then extracted with alkali in order to remove phenolic and acidic materials. The alcohols in the turpentine are then converted to non-volatile borates[7] by treatment with butyl borate, the hydrocarbon part of the turpentine being removed by vacuum distillation. The alcohols, liberated from the borate and steam distilled, are treated with phthalic anhydride and the unreacted tertiary alcohols (terpineols, etc.) are removed from the secondary alcohols, fenchol, etc. The crude acid phthalates are extracted with petroleum ether, then subjected to recrystallization for identification of the fenchol as the acid phthalate.

Preliminary tests using this procedure were made, using a synthetic mixture consisting of 1000 ml. of freshly distilled gum spirits to which had been added 0.07 g. of benzaldehyde, 1 g. fenchyl alcohol, and 0.03 g. furfural. No difficulty was experienced in obtaining positive tests for each of these compounds. The yield of 2,4 dinitrophenylhydrazone of benzaldehyde, and of acid fenchyl phthalate, were more than sufficient for the subsequent purification and recrystallization incident to the identification of these compounds.

Method of Testing:

The turpentine to be examined is steam distilled.

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[7] A. A. Kaufmann, French Patent No. 702,154, March, 1931.

Test a.—Approximately 250 ml. of the freshly distilled turpentine is extracted four times with 10-ml. portions of sodium bisulfite solution[8] and then washed with water. The combined bisulfite extracts and washings are then filtered through paper. The aldehyde-freed turpentine sample is reserved for test *b*. The bisulfite solution is made alkaline with sodium carbonate to liberate the aldehydes. The reaction mixture is then subjected to steam distillation and a total of about 100 ml. of steam distillate collected. If benzaldehyde is present, its odor will be perceptible in the steam distillate; also, if furfural is present it may be detected by testing a few drops of the distillate with aniline acetate.

The steam distillate is then extracted four times with 10-ml. portions of petroleum ether. The solvent separates as a cloudy layer, which may be cleared with a few grains of sodium chloride. The petroleum ether solution is washed twice with 10-ml. portions of water. After filtering through paper to remove mechanically held water, the solvent is evaporated. The oily residue is treated with 30 to 50 mg. of 2,4 dinitrophenylhydrazine and 3 to 5 ml. of glacial acetic acid. The reaction mixture is boiled for a few minutes and allowed to cool. If benzaldehyde is present, a solid promptly settles out. The dinitrophenylhydrazone is recrystallized from boiling acetone, then from boiling glacial acetic acid, and dried. The derivative should melt between 237 and 242 C. The melting point of pure benzaldehyde 2,4 dinitrophenylhydrazone is 241 to 242 C.

Test b.—The sample of turpentine from which the aldehydes had been removed is extracted with 2 *N* sodium hydroxide, washed with water, and steam distilled. The turpentine layer of the distillate is separated from the water and filtered through paper to remove mechanically held water. The alcohols contained in the sample are separated from the turpentine medium by treating in the following manner with butyl borate:

About 5 to 10 ml. of butyl borate are added to the turpentine in the distillation flask of a vacuum distillation assembly. The pressure is held at 50 to 60 mm. for 15 min., the reaction mixture is shaken occasionally, and the bath surrounding the distillation flask is heated to 70 C. during this period. The pressure is then lowered and the bath temperature increased to start rapid distillation. The bath temperature is gradually raised to 120 C. and the pressure reduced to 4 mm. and held there for 15 min. after the distillation has practically stopped. After 25 ml. of 2 *N* sodium hydroxide has been added to the borate remaining in the distillation flask, the liberated alcohols are steam distilled. The terpene alcohols are extracted from the steam distillate with petroleum ether and the petroleum ether is washed with water to remove traces of butyl alcohol. After filtering through paper, the solvent is evaporated. At this point the musty odor of fenchyl alcohol may be apparent. To the residue after evaporation of the petroleum ether is added one part by weight of phthalic anhydride and two parts by weight of benzene, and the whole heated under reflux on the steam bath for 40 to 48 hr.[9] The reaction mixture is cooled and shaken with an excess of 2 *N* sodium hydroxide and then extracted with ethyl

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[8] Sodium bisulfite solution was prepared by passing sulfur dioxide into a saturated solution of sodium carbonate. The sodium bicarbonate formed in the early part of the reaction redissolves, and the bubbling of sulfur dioxide is continued until saturated.

[9] This procedure has been found effective for preparing acid phthalates of secondary as well as primary alcohols, see S. Palkin, T. C. Chadwick and M. B. Matlack, "Composition and Fractionation of American Steam-Distilled Wood Turpentine," *Technical Bulletin No. 596*, U. S. Dept. Agriculture, Washington, D. C., November, 1937.

ether to remove tertiary alcohols. The alkaline water solution containing the acid phthalates of the secondary alcohols is then acidified with hydrochloric acid using Congo red as the indicator, and extracted with 75 to 100 ml. of benzene. After filtration of the benzene solution through paper to remove phthalic acid and traces of water, the solvent is evaporated. At this point the residue usually crystallizes, but if it does not immediately, it is covered with 5 to 10 ml. of petroleum ether and allowed to stand overnight to induce crystallization. When crystallization has taken place, the petroleum ether is decanted and the crystalline mass warmed to evaporate the adhering petroleum ether. The crystals are then dissolved in ethyl alcohol, the solution treated with decolorizing carbon, and filtered. The alcohol filtrate is then evaporated to about 5 to 10 ml., and an equal volume of water is slowly added. The crystals which thus form are recrystallized two or three times from 50 per cent ethyl alcohol, and finally dried. The melting point of fenchyl acid phthalate[10] is 169 to 170 C.

TABLE II.—RESULTS OF TESTS ON VARIOUS TUPENTINES.

Sample[a]	Kind of Turpentine[b]	Amount Used for Test, ml.	Benzaldedyde		Furfural Aniline Acetate Test	Fenchyl Alcohol	
			Test by Odor of "Aldehyde Distillate"	Melting Point of 2,4 Dinitrophenylhydrazone, deg. Cent.		Indicated by Odor of "Alcohol Distillate"	Melting Point of Isolated Acid Fenchyl Phthalate, deg. Cent.
No. 1	S.D.W.[b]	250	Positive	239 to 240	Positive	Indicated	169 to 170
No. 2	S.D.W.	500	Positive	241 to 242	Negative	Indicated	169 to 170
No. 3	S.D.W.	200	Positive	241 to 242	Positive	Indicated	169 to 170
No. 4	S.D.W.	200	Positive	239 to 240	Negative	Indicated	169 to 170
No. 5	S.D.W.	500	Positive	241 to 242	Negative	Indicated	169 to 170
No. 6	S.D.W.	250	Positive	241 to 242	Positive	Indicated	169 to 170
No. 7	S.D.W.	250	Positive	241 to 242	Positive	Indicated	169 to 170
No. 8	S.D.W.	250	Positive	241 to 242	Positive	Indicated	169 to 170
No. 9[c]	S.D.W.	250	Indicated	None obtained	Negative	Indicated	169 to 170
No. 10	S.D.W.	180	Positive	241 to 242	Negative	Indicated	169 to 170
No. 11	S.D.W.	80	Positive	241 to 242	Positive	Indicated	169 to 170
A	Gum	300	Negative	None obtained	Negative	Negative	None isolated
B	Gum	400	Negative	None obtained	Positive	Negative	None isolated
C	Gum	400	Negative	None obtained	Negative	Negative	None isolated
D	Gum	300	Negative	None obtained	Negative	Negative	None isolated
E	D.D.	250	Indicated	None obtained	Positive	Indicated	169 to 170
F	Sulfate	250	Indicated	None obtained	Negative	Negative	None isolated

[a] Sample numbers represent products from different producers.
[b] S.D.W. = steam-distilled wood turpentine, D.D. = destructively distilled turpentine.
[c] Does not conform with Society's specification for S.D.W.

In order to test the general applicability of the method, commercial samples of steam-distilled wood turpentine from several sources, and, for comparison, samples of gum spirits, of sulfate turpentine, and of destructively distilled wood turpentine were subjected to tests by the above method. The properties of these turpentines as determined by the Society's methods[11] are given in Table I. The results of test by the method described in this paper are shown in Table II.

As may be seen from Table II, all of the steam-distilled wood turpentine samples conforming to the Society's specifications showed the presence of

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[10] Harry Schmidt and Lee Schulz, "On Spatial Isomerism in the Fenchol Series," Annual Report on Essential Oils, Synthetic Perfumes, etc., 1935 English Edition, p. 93, Schimmel and Co., Miltitz near Leipzig.

[11] Standard Methods of Sampling and Testing Turpentine (D 233-36), 1936 Book of A. S. T. M. Standards, Part II, p. 744.

both benzaldehyde and fenchyl alcohol in sufficient quantity to permit isolation of derivatives. None of the samples of gum spirits gave any indication of these compounds.

Both destructively distilled and sulfate wood turpentine gave some indication by odor of the presence of benzaldehyde, but gave a negative test with the 2,4 dinitrophenylhydrazine, a delicate test for benzaldehyde. Fenchyl alcohol was isolated from the destructively distilled wood turpentine, but none was indicated in the sulfate turpentine.

The test for furfural, while included in the tabulated results, is not regarded as significant, since it has been found not only in steam-distilled wood turpentine, but also in gum and in destructively distilled wood turpentine, and does not occur consistently in any of these.

SUMMARY

Data have been presented on the fractionation and composition of American steam-distilled wood turpentine which show this turpentine to be decidedly different in composition from gum spirits. A chemical method has been described for the identification of steam-distilled wood turpentine, and for distinguishing it from gum spirits by tests for benzaldehyde and fenchyl alcohol—compounds shown to be present in commercial steam-distilled wood turpentine, but absent in gum spirits.

THE MECHANICAL PROPERTIES OF BALSA WOOD

By J. O. Draffin[1] and C. W. Muhlenbruch[2]

Synopsis

This report presents results of tests made on balsa wood ranging in density from 5.18 to 14.86 lb. per cu. ft. Properties investigated are the elastic limit, modulus of rupture and modulus of elasticity in bending; the elastic limit, ultimate strength and modulus of elasticity in compression parallel and perpendicular to the grain; column strength and cleavage. Studies are also reported on the strength of balsa under sustained or long-time bending loads.

The investigation shows that: (1) all of the mechanical properties of balsa which were studied vary with the density according to equations which have been developed, and (2) after allowance for the difference in density, the strength of balsa compares favorably, particularly for the higher densities, with the strength of such woods as white pine and redwood.

Balsa is a light-weight wood, from one-fifth to one-half as heavy as white pine. It is a second-growth wood, a native of Central and South America and the West Indies; in appearance it resembles the cottonwood tree. Because of its tropical habitat the tree grows rapidly, reaching a height of 36 ft. and a diameter of 25 in. in five years.

In the early years of growth, a very light pith-like wood is formed whereas in later years the tree develops a heavier type of wood. It is the light wood which is the subject of the present investigation.

At present balsa is used in many types of airplanes for various non-stress-carrying members such as outboard struts, fairing parts, flooring, partitions, panels, etc., in cabin construction. It is also used in recreational apparatus, in life preservers and as sound and heat insulation, both in the shredded and plank form. An airplane designer states that it is not used for stress-carrying parts in airplanes because not enough is known of its mechanical properties. Since there is such a small amount of information on balsa in engineering literature, the primary purpose of these tests was to provide data on the mechanical properties of this wood and thus make it possible to use it in the design of stress-carrying parts.

Materials and Specimens

The wood resembles white pine somewhat but has a silvery luster and, because of its uniform rate of growth, is devoid of annular rings. A planed surface has a streaked appearance similar to that seen in oak or chestnut. The wood is extremely light and porous and is easily injured or indented by blows or high local pressure perpendicular to the grain.

The material used in these tests was

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received as rough plank, 7 to 8 ft. long, 2 to 3 in. thick, and 7 to 8 in. wide, with an average moisture content of about five per cent. It was stored in the laboratory, which is warm and dry, and was cut into pieces of the desired size and shape and planed smooth. The density ranged from 5.18 to 14.86 lb. per cu. ft. or 0.0030 to 0.0086 lb. per cu. in., the latter being the unit used in presenting the data.

Specimens were prepared for tests in bending, compression parallel and perpendicular to the grain, cleavage and columns. The greater number of the bending tests were made on beams nominally $1\frac{1}{2}$ by $\frac{1}{2}$ by 20 in. which were tested flatwise with a concentrated load at the center of a span of 18 in. A few larger specimens 2 by 1 by 26 in., tested with a span of 24 in., were used for comparison with the smaller beams.

For tests in compression parallel to the grain, the specimens were 2 by 2 in. in cross-section and 6 in. long, while those for tests in compression perpendicular to the grain were 2 by 4 in. in cross-section and 2 in. in height. Columns ranged in length from 3 to 45 in. with cross-sections 1 by 1 in., $1\frac{1}{2}$ by $\frac{1}{2}$ in., and $1\frac{1}{2}$ by $1\frac{1}{2}$ in. Cleavage specimens were made according to the requirements of the A.S.T.M. Standard Methods of Testing Small Clear Specimens of Timber (D 143 - 27),[3] and were 2 in. wide with a minimum length of 2 in.

APPARATUS

Short-Time Tests.—Beam tests were made on a motor-driven beam-testing machine reading the load to 1 lb. All other tests were made on a hand-operated, screw-power machine reading the load to $2\frac{1}{2}$ lb. or on a hydraulic machine reading the load to 10 lb.

Readings of strain in compression parallel to the grain were taken over a gage length of 4 in., with a compressometer of the multiplying lever type fastened to the specimen by screws.

For compression perpendicular to the grain, the strain was read directly by means of two Ames dials which recorded the movement of the upper loading block on the specimen.

Columns were tested in a vertical position and were round-ended. The load was applied through plates at top and bottom, the plates rested on $\frac{3}{8}$-in. steel balls which effectively eliminated end restraint.

Long-Time Tests.—For the test of beams under sustained or long-time loads, a rack capable of holding 16 beams was built of two light I-beams placed 18 in. apart, center to center, and supported at three points. Small round rods were placed lengthwise on top of the I-beams and 18 in. apart to support the bearing plates on which the test beams rested. The loads consisted of concrete cylinders, made for the purpose and painted to avoid change in weight, hung at the mid-span of the $1\frac{1}{2}$ by $\frac{1}{2}$ by 20-in. beams. The loads were suspended from a yoke or loading saddle at the center of the span, the saddle resting on a $\frac{3}{8}$-in. steel ball which in turn rested on a bearing plate on the beam. Deflections of the beams were obtained with a detachable micrometer screw which measured the distance between the top of the loading saddles and the bottom of a small I-beam which was fastened to the rack slightly above the saddles.

TEST DATA AND DISCUSSION

It was necessary to handle the wood carefully in order to avoid injury to it, especially since the specimens tested were relatively small and an injury would have a considerable effect on the strength of the piece.

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[3] 1936 Book of A. S. T. M. Standards, Part II, p. 465.

The values of all properties were found to vary with the density of the specimens, as a rule the values being higher as the density increased.

Compression Parallel to Grain:

Properties Found from Stress-Strain Curves.—The tests for these properties were made on 70 specimens, with an attached compressometer to measure strain. The data were plotted as stress-strain curves from which the elastic limit and modulus of elasticity were obtained for the individual tests. The elastic limit was found to be between 65 to 95 per cent of the ultimate strength of the specimens with the screw holes from the compressometer. The strength of these specimens was 10 to 25 per cent, or an average of 15 per cent, less than the strength of the specimens without screw holes. Thus the elastic limit would be about 55 to 80 per cent of the ultimate strength of solid specimens, the values of which are plotted in Fig. 2. The values of the modulus of elasticity are plotted in Fig. 1, each plotted point being the average of 2 to 5 tests. The relation of the modulus of elasticity, E_c, in pound per square inch, to the density, σ, in pound per cubic inch, is given by the equation:

$$E_c = 346{,}000\ (560\sigma - 1)$$

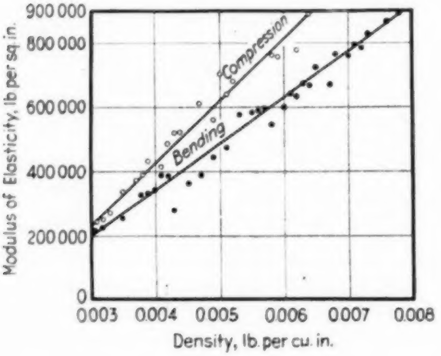

FIG. 1.—Relation of Modulus of Elasticity in Compression and Bending to Density.

The variation of the upper and lower limits from this equation is ±92,000 lb. per sq. in.

Compressive Strength.—A total of 84 pieces was tested for compressive strength, only the ultimate loads being observed. The results are shown in Fig. 2, each plotted point being the average of from 2 to 6 tests. The relation of compressive strength, S_c, in pound per square inch, to density, σ, is given by the equation:

$$S_c = 450\ (970\sigma - 1)$$

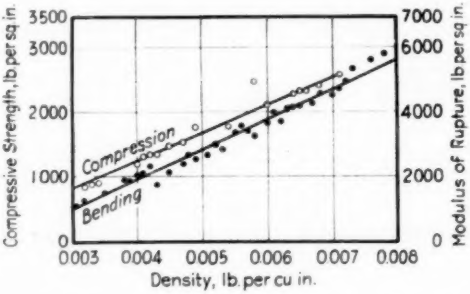

FIG. 2.—Relation of Compressive Strength and Modulus of Rupture to Density.

The variation of the upper and lower limits from this equation is ± 330 lb. per sq. in.

Compression Perpendicular to the Grain:

A total of 17 specimens was tested and load-strain readings taken well beyond the elastic limit. Stress-strain curves were plotted for each test and from these curves, which were very regular, the elastic limit and modulus of elasticity were found. The elastic limit ranged from 43 to 127 lb. per sq. in., with most values lying between 46 and 75 lb. per sq. in. The modulus of elasticity varied from 13,000 to 126,000 lb. per sq. in., with the larger number of values between 30,000 and 70,000 lb. per sq. in. The points were too scattered to warrant equations but without exception the higher densities had the higher values for both elastic limit and modulus of elasticity; the reverse, however, was not true since some high values of elastic limit and modulus of elasticity were found with low densities.

Beam Tests:

Short-Time Tests.—A total of 124 beams was tested in this series. The beams failed initially in compression and finally in tension. Those with the lower densities seemed more brittle and brash than those with higher densities. The larger beams, 2 by 1 by 26 in., gave somewhat lower values for all properties than the smaller beams, probably because the variation in density within any specimen was greater.

Load-deflection readings were taken and these data were plotted as curves from which the elastic limit, the modulus of rupture, and the modulus of elasticity were computed for each beam. The curves were similar in general shape to those ordinarily found for timber. The results obtained from individual curves were plotted as density-modulus of elasticity and density-modulus of rupture graphs and are shown in Figs. 1 and 2, respectively. Each plotted point for the beam tests represents, with one or two exceptions, the average of 3 to 6 tests. From the average line the relation of modulus of rupture, S_r, in pound per square inch, to density, σ, is given by the equation:

$$S_r = 1800\,(525\sigma - 1)$$

The variation of this equation from the upper and lower limits is ± 650 lb. per sq. in. The elastic limit was found to be between 76 and 94 per cent of the modulus of rupture.

The relation of modulus of elasticity in bending, E_b, in lb. per sq. in., to density, σ, is given by the equation:

$$E_b = 229{,}000\,(625\sigma - 1)$$

The variation of the upper and lower limits from this equation is ± 92,000 lb. per sq. in.

Long-Time Tests.—The length of the loading period is an important factor in beams, some beams of ordinary timber breaking at a load but little more than 50 per cent of that required to produce failure under short-time tests.[4] The modulus of rupture for any given density having been found by short-time tests, specimens of the same density, and therefore presumably of the same strength, were placed in the rack and loaded with various proportions of the load required to cause failure under the short-time test. The percentage used for different beams was from 20 to 90 with 66 per cent marking a critical value. The average time required for failure to occur in the case of all specimens loaded beyond 66 per cent of the short-time strength was 435 hr. Below 66 per cent no failures occurred.

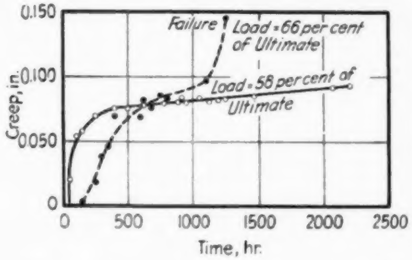

FIG. 3.—Characteristic Creep-Time Curves for Beams Subjected to Continued Loading.

After a beam had been placed on the rack and the load applied, deflection readings were taken each day or as often

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[4] H. F. Moore, "Textbook of Engineering Materials," Fifth Edition, p. 223, McGraw-Hill Book Co., Inc., New York City (1936).

as necessary. These deflection readings were then plotted against time. Since the deflections continued to increase with time, they were designated as creep, and the curves were called creep-time curves. Two such characteristic curves are shown in Fig. 3. One shows the creep-time relation for a specimen which failed after 1250 hr. under 66 per cent of the short-time ultimate load. The other is for a specimen which had not failed when loaded for 2200 hr. with 58 per cent of the ultimate load but which continued to deflect as the time increased. Many of these beams have been under load for 2500 to 4000 hr. and the deflection is still increasing. The humidity of the room affected the amount of deflection; during and immediately after a few damp days the deflection would increase rapidly and as the humidity again became normal the deflection would diminish.

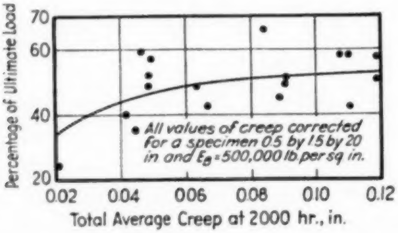

FIG. 4.—Relation of Load to Creep for Beams Subjected to Continued Loading.

All of the beams tested were adjusted to a modulus of elasticity and moment of inertia of an arbitrarily selected standard beam $1\frac{1}{2}$ by $\frac{1}{2}$ by 20 in. with a density of 0.0060 lb. per cu. in. By this adjustment of all beams to a common basis, it is possible to show the relationship between the percentage of the ultimate short-time load applied and the total creep for any given time, a period of 2000 hr. being used in Fig. 4. It will be noted that there is a large variation in the total creep for any given percentage of load, some specimens deflecting only 0.05 in. in 2000 hr., while others with the same proportionate load deflected 0.12 in. in the same time. The explanation of this difference seems to be that the density, and therefore the other properties, of balsa is variable to a high degree, not only between different specimens from the same plank but in different parts of the same specimen.

Column Tests:

The slenderness ratio, l/r, of the columns varied from 5 to 200, but since the columns were of different densities and consequently had different moduli of elasticity it was not possible to make an effective comparison on the basis of load and slenderness ratio alone. Accordingly, using a density of about 8.8 lb. per cu. ft., corresponding to a modulus of elasticity of 500,000 lb. per sq. in., as a basis, the slenderness ratio was changed to an equivalent value by varying the length inversely with the modulus of elasticity. This adjustment is in accordance with the Euler formula. The equivalent slenderness ratio is used in plotting the points in Fig. 5 where each point is the average of 4 to 5 tests. For slenderness ratios of 50 or more the average curve for the test results agrees

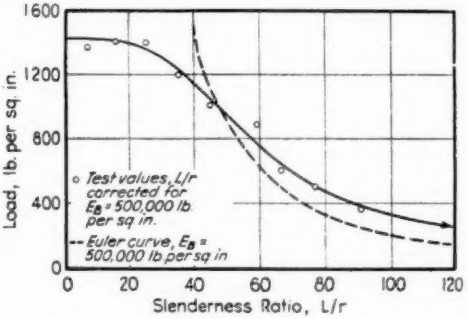

FIG. 5.—Relation of Load to Slenderness Ratio for Balsa Columns.

fairly well with the Euler curve based on round ends and an assumed modulus of elasticity of 500,000 lb. per sq. in. The average strength of these columns, based on a density of 8.8 lb. per cu. ft., may be expressed as follows:

For slenderness ratios less than 25, $P/A = 1400$ lb. per sq. in.;

between 25 and 80, $P/A = 1800 - 17\, l/r$;

in excess of 70 to 80, $P/A = \dfrac{1400}{1 + 0.00031\,(l/r)^2}$

Cleavage:

The ability of wood to resist splitting is sometimes of importance. Tests were made of this property using 35 cleavage specimens. The relation of cleavage strength, S_{cl}, in pound per linear inch, to density, σ, is shown in Fig. 6 and is expressed by the equation:

$$S_{cl} = 6\,(1430\sigma + 1)$$

Each plotted point in Fig. 6 represents 2 to 4 tests.

SUMMARY

The data presented in this paper with respect to balsa wood may be summarized as follows:

1. Balsa is a very soft wood and is easily indented or bruised; it is not, therefore, suitable for stress-carrying members which may be subjected to blows, rubbing against other parts, or abrasion. If used where such surface injury may occur, the member should be protected by some hard veneer surface.

2. All properties studied vary with the density, the numerical value of the property increasing as the density increases.

3. The average numerical values of the properties can conveniently be related to the density and appropriate simple equations constructed showing this relationship.

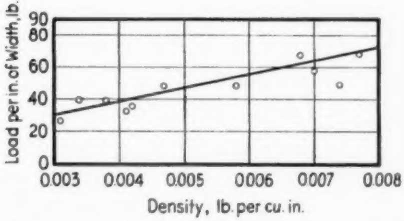

Fig. 6.—Relation of Cleavage Strength to Density.

4. There is the same relation between the properties of balsa and the properties of other light-weight woods, such as white pine, as there is between their densities.

5. When subjected to continued bending loads, balsa beams may be expected to fail when loaded to 60 per cent of the load required to cause failure in short-time tests.

6. Balsa may be safely used for stress-carrying members if careful consideration is given to the variation in density (or strength).

A THERMODYNAMIC AND COLLOIDAL INTERPRETATION OF PUBLISHED STUDIES ON THE CORROSION CRACKING OF STRESSED MILD STEEL IN WATER SOLUTIONS

By J. A. Tajc[1]

Synopsis

Assuming that at elevated temperatures solutions of sodium hydroxide and silica produce predominantly intercrystalline cracking in stressed mild steel, it is shown that the corrosion is not wholly electrochemical but that colloidal phenomena are involved. Thermodynamic calculations are made to show that the spontaneity of the corroding reactions of iron in water is not affected by the hydroxyl ion concentration and that the observed increase in the rate of corrosion with sodium hydroxide concentration is probably due to peptization. Reasons for the inadequacy of the hydrogen embrittlement theory in explaining caustic embrittlement are given and a colloidal explanation is suggested.

It is believed that silica precipitates the peptized iron oxide to protect the ferrite grains while permitting corrosion along highly stressed and probably cracked grain boundaries.

Concordance between the suggested explanation and experimental data in the literature is given as justification for the colloidal viewpoint.

Definition:

A special form of corrosion resulting in the cracking of steel has received considerable attention, especially from boiler users and pressure vessel fabricators. In the absence of a better definition and to distinguish it from previously identified forms of cracking due to time, temperature, stress and fatigue, the phenomenon was called "caustic embrittlement,"— caustic, because one of the earlier investigators believed caustic soda to be the controlling factor of the cracking. The several investigations[2, 3] disclose that embrittlement cracking is predominantly intercrystalline and occurs only when steel stressed beyond the yield point is immersed at elevated temperatures in solutions containing both caustic soda and silica. This is assumed to be the correct identification of the type of cracking discussed in this paper.

Corrosion of Iron:

The processes of iron and steel corrosion, whether they be classified as galvanic action, embrittlement, or pitting, are subject to the same physical and chemical principles. The spontaneity of any chemical reaction can be determined by the equilibrium constant formulated from the activities of the initial reactants and the final products

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[2] W. C. Schroeder and A. A. Berk, "Action of Solutions of Sodium Silicate and Sodium Hydroxide at 250 C. on Steel Under Stress," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 120, p. 387 (1936).

[3] S. W. Parr and F. G. Straub, "Embrittlement of Boiler Plate," *Bulletin 177*, University of Illinois Engineering Experiment Station, p. 29 (1928).

without regard to the intermediate or transition stages. Great importance is therefore attached to knowing what molecules are being attacked and what the end products of the attack may be.

Berl and van Taack[4,5] report that at boiler temperatures the end product of corrosion of iron in oxygen-free water is Fe_3O_4 containing excess FeO. Smith's[6] experiments indicate that when electrolytic iron is heated in air above 200 C. (392 F) either γ-Fe_2O_3 or possibly Fe_3O_4 is formed. The critical and exhaustive review of iron oxides by Welo and Baudisch[7] leads to similar conclusions. Other investigators[8, 9] observed that the film on air-oxidized iron consisted of successive layers of iron in various states of oxidation: FeO adjacent to the metal, then a layer of Fe_3O_4 and an outermost layer of Fe_2O_3. It is very probable that an analogous multilayered film may be found on boiler surfaces, especially in view of the fact that Fe_3O_4 free from other Fe-O-H_2O compounds is very difficult to prepare and that FeO decomposes below 570 C. (1060 F.) into Fe and Fe_3O_4.

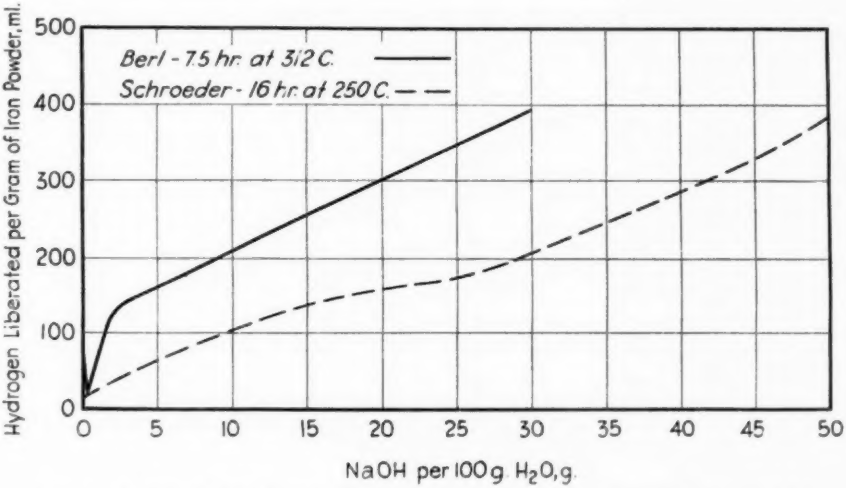

FIG. 1.—Effect of NaOH upon Rate of Corrosion of Iron Powder as Measured by Hydrogen Evolution.

The attempts to explain embrittlement cracking have resulted in considerable speculation concerning the existence, the method of formation, and the stability of this oxide film in sodium hydroxide solutions. It has been suggested[10] that sodium hydroxide reacts directly with the iron to yield a film of magnetic iron oxide according to the equation:

$$3Fe + 4OH^- \rightarrow Fe_3O_4 + 2H_2 \ldots (1)$$

which upon completion and a balancing of charges becomes

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[4] E. Berl and F. Van Taack, "The Protective Action of Sodium Sulfate on the Attack of Ingot Iron by Alkalies and Salts Under High Pressure," *Archiv für Wärmewirtschaft und Dampfkesselwesen*, Vol. 9, p. 165 (1928).

[5] E. P. Partridge and W. C. Schroeder, "Embrittlement of Boiler Steel," *Metals and Alloys*, Vol. 6, pp. 145, 187, 253 311, 355 (1935).

[6] N. Smith, "Structure of Thin Films of Metallic Oxides and Hydrates," *Journal*, Am. Chemical Soc., Vol. 58, p. 173 (1936).

[7] L. A. Welo and O. Baudisch, "Active Iron," *Chemical Reviews*, Vol. 16, p. 45 (1934).

[8] R. M. Bozorth, "Structure of a Protective Coating of Iron Oxides," *Journal*, Am. Chemical Soc., Vol. 49, p. 969 (1927).

[9] D. W. Murphy, W. P. Wood and W. E. Jominy, "Scaling of Steel at Elevated Temperatures by Reaction with Gases and the Properties of the Resulting Oxides," *Transactions*, Am. Soc. Steel Treating, Vol. 19, p. 193 (1931).

[10] F. G. Straub, "Embrittlement in Boilers," *Bulletin 216*, University of Illinois Engineering Experiment Station, p. 93 (1930).

$$3Fe + 4NaOH \rightarrow Fe_3O_4 + 2H_2 + 4Na \quad\ldots\ldots(2)$$

This reaction as well as that proposed by Thiel[11] would produce sodium metal by the reaction of iron with sodium hydroxide, for which there is no experimental evidence.

The influence of sodium hydroxide upon the corrosion of boiler steel was investigated by both Berl[5] and Schroeder,[12] who placed powdered steel in a bomb at constant temperature and different concentrations of NaOH. They ascertained the extent of the corrosion, or equivalent oxidation, by observing the increase in bomb pressure due to the hydrogen evolved and by determining the increase in the oxygen content of the powder. The results of the two investigations are compared in Fig. 1, where Berl's data for increase in the oxygen content of the powder is expressed in terms of equivalent hydrogen evolved, on the basis that during the reaction, water is the sole source of both hydrogen and oxygen. It will be noted that while the tests are not strictly comparable because of different operating conditions, they both indicate that the attack of solutions of sodium hydroxide upon steel increases with concentration.

There is a widespread belief that the sodium hydroxide promotes the corrosion by dissolving the protective layer of Fe_3O_4 according to the equation:

$$Fe_3O_4 + 4NaOH \rightarrow Na_2FeO_2 + Na_2Fe_2O_4 + 2H_2O \quad\ldots\ldots(3)$$

This explanation postulates the existence of sodium ferrite and ferrate in water solution at boiler temperatures, and presumably takes no cognizance of the fact that the ferrous and ferric hydroxides have been reported insoluble in solutions of sodium hydroxide[13,14] and that the ferrate and ferrite hydrolyze in water to yield free sodium hydroxide. In view of these and other conflicting and chemically inconsistent opinions, it is unquestionably more logical to consider the corrosion processes in the light of fundamental and incontrovertible chemical data.

Assuming that the corrosion product is Fe_3O_4 and that the OH ion concentration is a controlling factor for the corrosion of iron in sodium hydroxide solutions, then

$$3Fe + 4OH^- \rightarrow Fe_3O_4 + 2H_2 + 4\,\ominus \quad\ldots\ldots\ldots\ldots(4)$$

is the equation of the corroding reaction. It is believed that the four electrons liberated in Eq. 4 are used up as follows:

$$4\,\ominus + 4H^+ \rightarrow 2H_2 \quad\ldots\ldots(5)$$

adding Eqs. 4 and 5

$$3Fe + 4OH^- + 4H^+ \rightarrow Fe_3O_4 + 4H_2 \quad\ldots\ldots(6)$$

Then without speculating as to what the intermediate reactions may be, the equilibrium constant for Eq. 6 may be formulated:

$$K_1 = \frac{a_{Fe_3O_4} \cdot a^4_{H_2}}{a^3_{Fe} \cdot a^4_{OH^-} \cdot a^4_{H^+}} = \frac{a_{Fe_3O_4} \cdot a^4_{H_2}}{a^3_{Fe}\,(a_{OH^-} \cdot a_{H^+})^4} \quad\ldots\ldots(7)$$

For the reaction $H_2O \rightarrow H^+ + OH^-$ the equilibrium constant for concentrated solutions is

$$K_2 = \frac{a_{OH^-} \cdot a_{H^+}}{a_{H_2O}} \text{ or } a_{OH^-} \cdot a_{H^+} = a_{H_2O} \cdot K_2 \quad\ldots\ldots\ldots\ldots(8)$$

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[11] A. Thiel, "Speisewasserplege," Vereinigung der Grosskesselbesitzer, Berlin (1925).

[12] W. C. Schroeder, Progress Report No. 10 to Joint Research Committee on Boiler Feedwater Studies, Private communication (1937).

[13] J. W. Mellor, "Treatise of Inorganic and Theoretical Chemistry," Vol. 13, Part II, pp. 720, 930, Longmans, Green and Co., New York City (1934).

[14] A. B. Prescott and O. C. Johnson, "Qualitative Chemical Analysis," Seventh Edition, p. 155, D. Van Nostrand Co., New York City (1916).

Substituting for $a_{OH^-} \cdot a_{H^+}$ in Eq. 7 and combining K_2 and K_1

$$K_3 = \frac{a_{Fe_3O_4} \cdot a^{4}_{H_2}}{a^{3}_{Fe} \cdot a^{4}_{H_2O}} \quad \ldots\ldots\ldots (9)$$

Equation 9 is a complete and logical conclusion if Eqs. 4 and 5 are true. It implies that the equilibrium equation for the corrosion of iron in sodium hydroxide solutions is controlled not by the OH ion activity, but by the H_2O activity.

Assuming end products other than Fe_3O_4 does not alter the direction of the reaction. If the iron goes into solution at room temperature according to the equation:

$$Fe + 2H_2O \rightarrow Fe(OH)_2 + H_2 \quad \ldots\ldots (10)$$

equilibrium will not be established until the pressure of hydrogen is approximately 60 atms. or 880 lb. per sq. in.[15, 16] Furthermore, at 242 C. (470 F.) the partial pressure of O_2 due to the dissociation of water into hydrogen and oxygen is approximately 10^{30} times as great as that just sufficient to make all of the following reactions spontaneous.[17, 18, 19]

$$Fe + \tfrac{1}{2}\, O_2 \rightarrow FeO \qquad \Delta F_{515} = -54{,}000 \text{ cal.} \ldots (11)$$

$$2Fe + \tfrac{3}{2}\, O_2 \rightarrow Fe_2O_3 \qquad \Delta F_{515} = -162{,}000 \text{ cal.} \ldots (12)$$

$$3Fe + 2\, O_2 \rightarrow Fe_3O_4 \qquad \Delta F_{515} = -223{,}500 \text{ cal.} \ldots (13)$$

Since under boiler operating conditions it is quite impossible to prevent the H_2 from escaping in reactions 6 and 10, and equally impossible to prevent the dissociation of water into oxygen and hydrogen thus allowing reactions 11, 12 and 13 to continue, we must conclude that the only way to decrease the spontaneity of the corroding reactions in water solutions is to exclude the water from contact with the iron.

Corrosion of Metals and Metallic Compounds:

For any metal below hydrogen in the electromotive series, the general equation for corrosion might be written (using the letter "M" to represent the metal) as follows:

Metal + Water → Metal Oxide (or Hydroxide) + Hydrogen

or

$$M + xH_2O \rightarrow M(OH)_x + \frac{x}{2}\, H_2 \quad \text{(for hydroxide formation)}. (14)$$

for which

$$K_4 = \frac{a_{M(OH)_x} \cdot a^{\frac{x}{2}}_{H_2}}{a_M \cdot a^{x}_{H_2O}} \quad \ldots\ldots (15)$$

or

$$2M + xH_2O \rightarrow M_2O_x + xH_2 \text{ (for oxide formation)} \ldots\ldots\ldots (16)$$

for which

$$K_5 = \frac{a_{M_2O_x} \cdot a^{x}_{H_2}}{a_M \cdot a^{x}_{H_2O}} \quad \ldots\ldots (17)$$

It will be noticed, for both these general equations, that if the hydrogen is permitted to escape, the reactions will continue to completion. Hence the only way to prevent the corrosion is to reduce the activity of water to zero at the metal surface. This can only be ac-

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[15] J. C. Warner, "Organic Type Inhibitors in the Acid Corrosion of Iron," *Transactions*, Am. Electrochemical Soc., Vol. 56, p. 287 (1929).

[16] L. W. Elder, "A Comparison of Certain Hydrogen Ion Indicator Electrodes in the Presence of Ferric Iron," *Transactions*, Am. Electrochemical Soc., Vol. 57, p. 393 (1930).

[17] M. Randall and M. Frandsen, "Determination of the Free Energy of Ferrous Hydroxide from Measurements of Electromotive Force," *Journal*, Am. Chemical Soc., Vol. 54, p. 40 (1932).

[18] J. Chipman and D. W. Murphy, "Free Energy of Iron Oxides," *Industrial and Engineering Chemistry*, Vol. 25, p. 319 (1933).

[19] International Critical Tables, Vol. 7, p. 232 (1930).

complished by the exclusion of the water from the metal surface; a conclusion already reached for iron and water. Similarly, for any metallic compound, the general reaction may be written:

$$\mathrm{M}_y\mathrm{A}_x + xy\mathrm{H_2O} \rightarrow y\mathrm{M(OH)}_x + x\mathrm{H}_y\mathrm{A} \quad \ldots\ldots\ldots\ldots (18)$$

for which

$$K_6 = \frac{a^y_{\mathrm{M(OH)}_x} \cdot a^x_{\mathrm{H}_y\mathrm{A}}}{a_{\mathrm{M}_y\mathrm{A}_x} \cdot a^{xy}_{\mathrm{H_2O}}} \quad \ldots\ldots (19)$$

The former reactions (Eqs. 14 and 16) take place at the ferrite grains, the latter (Eq. 18) involving a metallic compound very likely takes place at the grain boundaries of the metal. From the general Eq. 18, the effect of sodium hydroxide in promoting the reaction may be inferred; it would neutralize the acid and reduce its activity nearly to zero in the equilibrium equation.

The reactions written above explain only that mild steel should be attacked in water solutions. They provide no explanation for the selective attack on the grain boundaries nor do they explain the contribution of silica and of caustic toward producing the cracking. We must conclude that an adequate explanation of embrittlement cannot be made by studying the corrosion of iron alone.

Hydrogen Embrittlement:

It has been suggested that caustic embrittlement may be a special case of hydrogen embrittlement. If this is true, then the phenomenon is essentially concerned with the diffusion of hydrogen through the steel and is similar to the embrittlement produced during the acid pickling of steel. The hydrogen, as has been previously pointed out, is generated according to Eqs. 14 or 16 during the reactions between iron and water to form either the oxide or hydroxide.

The literature did not settle until recently the nature of the diffusion process of hydrogen through iron, whether through the lattice or along the grain boundaries.[20, 21, 22, 23, 24, 25] However, the work of Smithells and Ransley[20] shows that for hydrogen through iron, only lattice diffusion is involved. Ham[21] extended the work to determining diffusion rates as well as the effect of impurities on the diffusion process. The observation of Korber and Ploum[22] that pure iron does not absorb hydrogen as readily as impure iron is supported by Edwards[23] and Morris[24] who noted that hydrogen diffuses through steel but not so readily through oxides and inclusions contained in it. Some explanation of this behavior may be found in a recent report by Mehl,[25] where the general equation of diffusion in terms of atomic constants is given as:

$$D = \frac{Q}{Nh}\delta^2 e^{-\frac{Q}{RT}}$$

where D = diffusion coefficient,
Q = heat of diffusion,
N = Avagadro's constant,
h = Planck's constant, and
δ = interatomic distance.

It is to be noted that if the density is lower, the interatomic distance must of necessity be greater and the gases diffuse far more readily. This would account for the greater diffusion in impure iron, and may in a measure

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[20] C. J. Smithells and C. E. Ransley, "The Diffusion of Gases Through Metals," *Proceedings*, Royal Soc. (London), Vol. 150, p. 172 (1935).

[21] W. R. Ham, "The Diffusion of Hydrogen Through Nickel and Iron," Presented at October, 1936, meeting, Am. Soc. Metals, issued as preprint but not published.

[22] F. Korber and H. Ploum, "Über die Aufnahme des Wasserstoffs Durch Eisen," *Zeitschrift für Electrochemie und angewandte physikalische Chemie*, Vol. 39, p. 252 (1933).

[23] C. A. Edwards, "Pickling: or, the Action of Acid Solutions on Mild Steel and the Diffusion of Hydrogen Through the Metal," *Journal*, Iron and Steel Inst., Vol. 110, p. 9 (1924).

[24] T. N. Morris, "Diffusion of Hydrogen Through Mild Steel Sheet During Acid Corrosion," *Journal*, Soc. Chemical Industry (London), Vol. 54, p. 7 (1935).

[25] R. F. Mehl, "Diffusion in Solid Metals," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 122, p. 11 (1936).

account for that along the grain boundaries of the steel, for the compounds of iron have in general a lower density than does the pure iron itself. (The rule of phase indicates that during solidification of steel, the last portion to crystallize contains the greatest quantity of solute—impurities—in solid solution.) Diffusion through an inclusion or an oxide may be an entirely different problem, because of the change in the value of Q, the heat of diffusion, even though the interatomic distance is the same or even greater. Hydrogen embrittlement and caustic embrittlement are similar[26] in several respects. They are both characterized by a predominantly intercrystalline tensile failure (coincidental to a marked decrease in the ductility) and by the ability of uncracked but embrittled specimens to recover their ductility upon being exposed to the air for a long time or upon heating for a short time. In other respects the comparison fails. Hydrogen generated in acid solutions embrittles unstressed steel, while it is generally agreed that relatively high stresses are required to embrittle steel immersed in sodium hydroxide solutions. In the diffusion equation, there is no provision for a stress factor unless it be incorporated in some way either in the heat of diffusion, Q, or in the change of the interatomic distance, δ. In addition, hydrogen embrittlement is accompanied by no appreciable reduction in the tensile strength of the steel such as appears characteristic of caustic embrittlement.

For the action of silica there appears to be no explanation; corrosion and hydrogen evolution proceed both in the presence and in the absence of silica, while Schroeder[12] has demonstrated that there is no correlation between the overall reaction (as measured by hydrogen evolution) and the rate of cracking.

From the foregoing, it appears that embrittlement cannot be explained by the hydrogen embrittlement hypothesis. A tenable theory should not only identify the action of sodium hydroxide, of silica and of stress individually but also harmonize the derived conclusions with the experimental evidence, that all three are necessary for the characteristic intercrystalline failure.

Influence of Sodium Hydroxide and Silica:

Assuming that in sodium hydroxide solutions at 250 C. a piece of unstressed steel starts to corrode according to Eqs. 14 or 16 and 18, then no cracks are evident; the grains as well as the grain boundaries are removed at a very slow rate. Should the grain boundaries not be attacked, there still would be no change in the surface appearance because of the great predominance of the grain areas. Berl and Schroeder's data in Fig. 1 indicate that sodium hydroxide accelerates the corrosion of iron, whereas Eqs. 14, 16, and 18 indicate that sodium hydroxide is neither an active ingredient of the corrosion process nor a factor in determining the stability of the resulting iron oxide. These diverse conclusions may be reconciled without rationalization by evidence that KOH, NaOH, and Na_3PO_4 readily peptize iron oxide to yield a colloidal solution, and that an increase in the peptization follows an increase in the alkalinity.[7,27,28,29,30,31] Apparently the sodium hydroxide dis-

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[26] A. E. White and R. Schneidewind, "Fractures in Boiler Metal," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 53, p. 193 (1931).

[27] A. von Buzagh, "A Method of Preparing Colloid-Soluble Iron Oxide and the Properties of Its Hydrosol," *Kolloid-Zeitschrift*, Vol. 66, p. 129 (1934).

[28] A. Dumanskii and V. M. Simonova, "The Method of the Triangular System of Coordinates in Colloid Chemistry," *Journal of General Chemistry*, (U. S. S. R.), Vol. 1, p. 209 (1931).

[29] S. H. Carsley, "The Reduction of Alkali Nitrates by Hydrous Ferrous Oxide," *Journal of Physical Chemistry*, Vol. 34, p. 178 (1930).

[30] G. H. Ayers and C. H. Sorum, "A Study of the Influence of Hydrolysis Temperature on Some Properties of Colloidal Ferric Oxide," *Journal of Physical Chemistry*, Vol. 34, p. 875 (1930).

[31] F. Hazel and G. H. Ayers, "Migration Studies with Ferric Oxide Sols," *Journal of Physical Chemistry*, Vol. 35, p. 2930 (1931).

integrates the protective oxide film by peptization thus promoting further corrosion of the underlying ferrite. For protecting the ferrite against attack, we have a choice of two methods: by reversing the spontaneity of Eqs. 14 or 16, or by preventing the peptization of the protective oxide. From Eq. 9 we know that reversing the direction of the ferrite corrosion is a remote possibility; by contrast the peptization is easily arrested by a suitable ion or colloid, the nitrates, sulfates, chromates, iodates, and chlorides being characteristic examples of the ionic group, while stannic oxide, selenium, platinum, and silica are examples from the colloidal group.

The inclusion of silica among the protectors of ferrite appears anomalous to the evidence[2] that small quantities of silica greatly accelerate the failure and cracking of stressed mild steel in sodium hydroxide solutions—a steel which is principally ferrite. When silica protects the ferrite grains, it would follow that the corrosion is confined to the impurities and the grain boundaries. Since the boundaries make up only a very minute portion of the total volume of the steel, a very small amount of corrosion would cause considerable penetration; cracks naturally follow. But, under these conditions, we find that neither grain boundary corrosion nor cracking is evident unless the steel is stressed near its yield point.

Influence of Stress:

Stress increases markedly the chemical activity of the steel as a whole. The increase in the activity of the ferrite grains does not disturb their rate of corrosion; that is already established by the rate of peptization of the oxide whose activity remains unchanged.

The unprotected grain boundaries tend to disintegrate according to Eq. 18, but it appears that high stresses are necessary to raise the reactants to a plane of chemical activity where the corrosion is spontaneous. Any slight initial boundary disintegration gives rise to stress concentration; the grain boundary, due to its brittle nature, may crack and promote further disintegration by exposing a much larger area to attack. It is probable that stress makes the reactions spontaneous and simultaneously produces submicroscopic cracks in which corrosion takes place. Should the corrosion products be unable to escape from the confines of the crack the reaction may soon be stifled; but not so the cracking, for if there is a volume increase during the corrosion process (as is frequently the case) then tremendous bursting forces may be generated in the crack. Thus the course of the crack is altered and stress gradients, at variance with those externally applied, may exist in the body of the metal. In addition, the stress intensifies the ever-present anodic and cathodic areas such as Lochte and Paul[32] found on an electrode composed of annealed pure iron, 99.859 per cent Fe and 0.026 per cent carbon having an area of 0.00011 sq. cm. If variations in atomic activity are found in iron of this purity, then anodic areas exist in ordinary impure and unannealed steel where the crystalline discontinuities, the grains of high surface energy and the unequal distribution of stress in the aggregate, all encourage unequal chemical activity. The danger of such areas lies in the generated potential which induces the migration of ions and particles that otherwise establish chemical equilibrium and gives a tremendous impetus to reactions which naturally stifle themselves.

Such a mechanism is quite probable. A crack or a slip fault in ferrite to be visible under the most powerful micro-

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[32] H. L. Lochte and R. E. Paul, "Electrochemical Behavior of Iron in Corrosion Cells," *Transactions*, Am. Electrochemical Soc., Vol. 64, p. 155 (1933).

scope must have a magnitude of several hundred atomic diameters, and water molecules have ample access to the faces of a crack long before that action can be visually noted. Freshly cracked faces are unquestionably more reactive than the adjacent metal. The resulting corrosion and penetration, preceded by cracking, proceed very rapidly. Similar submicroscopic cracks are naturally to be expected in the extremely brittle grain boundaries whenever, as a result of stress, adjustments and deformations take place in the plastic ferrite matrix, for which yield points as low as 5000 lb. per sq. in. have been reported.[33, 34, 35, 36] It is apparent that under similar loads the ferrite is deformed without separation, while the more brittle grain boundary may develop cracks along which corrosion continues. The action of stress may be summarized as follows: it increases the activity of the grain boundaries, it produces anodic areas, and finally it results in submicroscopic cracks which become focal points for the corrosion.

The Colloidal Explanation:

The previously suggested mechanism by which silica may influence the corrosion processes of steel in sodium hydroxide solutions, requires some amplification and considerable qualification. SiO_2 and Na_2O mixtures have various commercial designations, being called silicates, water glass and colloidal silica. It is at present agreed that only two silicates of soda exist, Na_2SiO_3 and $NaHSiO_3$. Any other ratios of Na_2O to SiO_2 are examples of colloidal electrolytes.[37, 38, 39, 40, 41] Britton [42, 43] points out that Na_2SiO_3 is not a true salt; consequently, it is quite probable that at 250 C. even the true silicates are hydrolyzed according to the equation:

$$\begin{aligned} \mathrm{Na_2SiO_3} + \mathrm{2H_2O} &\rightarrow \mathrm{2NaOH} \\ &+ \mathrm{SiO_2 : H_2O} \\ &\qquad \Big\downarrow \text{heat} \\ &\mathrm{SiO_2} + \mathrm{H_2O} \end{aligned}$$

The product of such hydrolysis in sodium hydroxide solutions is a negative hydrosol of SiO_2 stabilized by NaOH.

The colloids of iron oxide prepared in sodium hydroxide solutions have been reported negative at pH 8.6 and above, but the recent investigation of Hazel and Ayers[31] indicate that pH alone does not determine the *iso*-electric point of an iron oxide sol. They have shown the positive sol to be stable at ordinary temperatures above a pH 9 while Welo and Baudisch give dehydration curves for the sol up to 250 C. It is therefore conceivable that the positive iron oxide sol could exist at the higher pH and temperature of the boiler.

A negatively charged colloid and a positively charged colloid of approximately equal charge, mutually precipitate each other.[31, 45, 46] If either is in excess, it may stabilize the other. Dur-

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[33] H. S. Rawdon, "Strain Markings in Mild Steel Under Tension," National Bureau of Standards *Journal of Research*, Vol. 1, p. 467 (1928).

[34] H. J. Gough, "Crystalline Structure in Relation to Failure of Metals—Especially by Fatigue," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 3 (1933).

[35] C. A. Edwards and L. B. Pfiel, "The Tensile Properties of Single Iron Crystals and the Influence of Crystal Size Upon the Tensile Properties of Iron," *Journal*, Iron and Steel Inst., Vol. 112, p. 79 (1925).

[36] "Plastic Deformation of Iron," Am. Soc. Metals Handbook, p. 392 (1933).

[37] H. B. Weiser, "Hydrous Oxides," p. 196, McGraw-Hill Book Co., Inc., New York City (1926).

[38] W. Stericker, "The Relation of Structure to Free Alkali in Sodium Silicate Solutions," *Chemical and Metallurgical Engineering*, Vol. 25, p. 61 (1921).

[39] Kenkyo Inaba, "Hydrosol of Silicic Acid—III," *Scientific Papers*, Inst. Physical and Chemical Research (Tokyo), Vol. 15, p. 277 (1931).

[40] R. W. Harman, "Aqueous Solutions of Sodium Silicates," *Journal of Physical Chemistry*, Vol. 32, p. 44 (1928).

[41] J. S. Vail, "Soluble Silicates in Industry," pp. 17–71 Chemical Catalog Co., New York City (1928).

[42] H. T. S. Britton, "Hydrogen Ions," p. 357, Chapman and Hall, (London) (1932).

[43] *Ibid.*, p. 466.

[44] C. S. Hitchen, "A Method for the Experimental Investigation of Hydrothermal Solutions," *Transactions*, Inst. Mining and Metallurgy, Vol. 44, p. 255 (1934–1935).

[45] E. K. Rideal, "Surface Chemistry," p. 194, Cambridge University Press (1930).

[46] *Ibid.*, p. 272.

ing the corrosion of steel the concentration of the iron oxide sol is never very great, consequently it can never have a stabilizing influence. On the other hand, a concentration of the silica in excess of that required to produce mutual co-precipitation may have a peptizing influence upon the ferric oxide-silica precipitate. This may account for the optimum concentration of silica to cause embrittlement cracking. It must be remembered that the argument is equally valid if the process is not one of neutralization but of pure adsorption, for the two processes are frequently indistinguishable.

The previously mentioned experiments by Schroeder[12] upon the corrosion of iron powder in sodium hydroxide solutions were extended to include the influence of silica. His curve relating the concentration of silica to the rate of hydrogen evolution is given in Fig. 2. The experimental conditions were the same as for the data in Fig. 1 except that the sodium hydroxide concentration remained uniform at 25 g. per 100 g. of water.

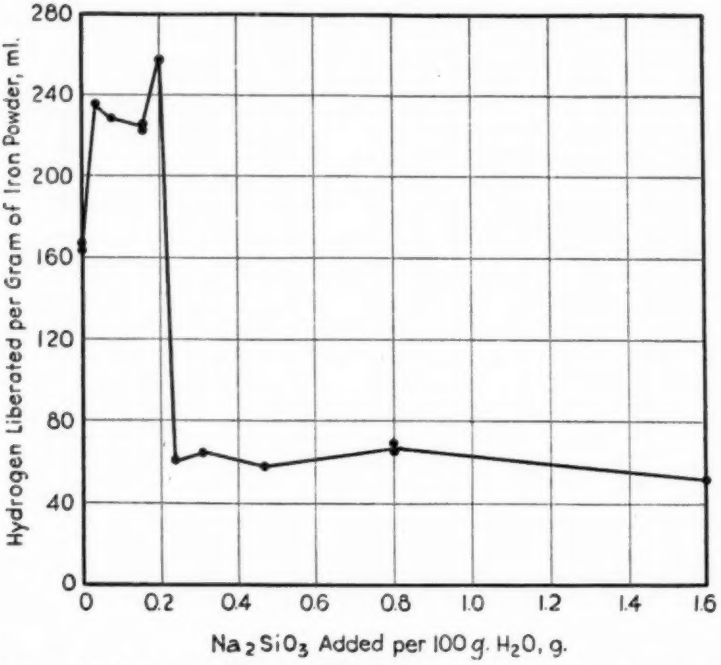

FIG. 2.—Effect of Na_2SiO_3 upon the Corrosion Rate of Iron Powder in Solution Containing 25 g. NaOH per 100 g. H_2O. Each test run for 16 hr. at 250 C. Data by W. C. Schroeder.

The irregularities in the curve of Fig. 2 may be readily explained on the basis of the colloidal hypothesis. Small additions of silica increase the corrosion rate because the silicate ion is also an effective peptizing agent, this may account for the rise in hydrogen evolution of Fig. 2. However, the solubility of silica is very low and decreases with a rise in temperature[44] so that in concentrations above saturation, the silica mixes with caustic soda but exists primarily as a colloid.[37, 38, 39, 40, 41, 42, 43]

As the concentration of silica slowly increases beyond its saturation value, mutual coagulation starts between the iron oxide sol and the silica sol; precipitation does not occur until a critical concentration is reached, so that while the number of "free" iron oxide particles decreases, the corrosion rate remains unchanged until the flocculating concentration of silica sol is reached, at which point the entire mass, the silica and the iron oxide, flocculates as an amorphous precipitate. (The term "free" has been coined to distinguish the peptized iron oxide particle from similar particles that have been adsorbed on the silica but which still remain in colloidal suspension.) Increasing the silica concentration beyond the flocculating point has very little effect upon the over-all corrosion; the grain boundaries corrode and crack, while the grains remain protected. However, a large excess of silica may be adsorbed at the grain boundaries by the precipitate on contiguous grains so that a protective effect may be produced and the cracking may be diminished. When this condition of the surface is attained, the counter diffusion processes of hydrogen and water through the iron-silica precipitate are the controlling factors in the corrosion. The colloidal explanation of the corrosion and cracking is supported by the fact that the region of decreasing hydrogen evolution in Fig. 2—the region of flocculation—is the same region that had been previously identified as one of maximum embrittlement. It confirms our belief that cracking occurs whenever the corrosion is selective and the mass of the metal is protected.

Schroeder[12] reported that in the region of increasing hydrogen evolution the iron oxide crystals on the powder increased in size with increasing silica concentrations. This is easily understood because only the "free" iron oxide particles can serve as crystallization nuclei and as their number decreases the crystals naturally grow larger. It must be remembered that crystallization from colloidal solution is not an unusual phenomena and that a colloid such as silica may change the crystallographic form as well as the particle size, and consequently petrographic and chemical analysis may be very misleading as to the structural compositions of the precipitate.[45, 46]

It is admitted that the colloidal explanation has limitations and that not all the questions can be answered with the available data. There appear to be no records of specific tests involving the relative corrosion of iron and its grain boundaries; hence, the conclusions have been based almost entirely on the experiments of investigators who, with few exceptions, have no interest either in corrosion or in "caustic embrittlement." The interpretation of these data and their application to embrittlement is entirely personal. But despite the shortcomings of the suggested mechanism, it is concluded that the action of silica is colloidal and that the solution of the embrittlement problem may lie in the colloidal field; probably among the organo-hydrophilic colloids.

Acknowledgments:

The author is deeply indebted to W. C. Schroeder, the Joint Research Committee on Boiler Feedwater Studies, and the New Brunswick Station of the U. S. Bureau of Mines for permission to use data on the corrosion of iron powder; and to D. S. McKinney, of the Carnegie Institute of Technology, who suggested the investigation and rendered valuable assistance in preparing the paper.

DISCUSSION

Mr. Thomas J. Finnegan[1] (*presented in written form*).—The paper by Mr. Tajc supports the conclusions of Schroeder and his coworkers that the effect of high concentrations of sodium hydroxide is to destroy the protective oxide film on the metal. He has gone a step further in postulating a mechanism for the removal of the film, namely, peptization by the sodium hydroxide. It will be highly interesting to follow the subject from now on, as Mr. Tajc's theory will certainly receive experimental study.

There seems to be, however, one or two matters which as yet are not quite convincing. For instance, if we consider that sodium hydroxide does not enter into the fundamental corrosion reaction but plays a secondary part in removing the film, we must still explain the evolution of hydrogen in its presence.

In an aqueous reaction the fundamental step is the effect of the solution pressure of iron:

$$Fe + 2H^+ \rightarrow Fe^{++} + H_2$$

The activity of water as described in the paper must embrace the activity of hydrogen and hydroxide ions. The presence of NaOH will lower the hydrogen-ion activity to some very low value. Would not this be expected to lower the rate of hydrogen evolution? According to Fig. 1 of the paper, however, the hydrogen evolution is high at concentrations of NaOH of such magnitudes that the hydrogen-ion concentrations have been reduced to practically insignificant values. Furthermore, increasing the NaOH until the solution is syrupy increases the hydrogen evolution. If this condition can be explained solely on the basis of the fundamental reaction of iron and water, it should be done. Offhand it would appear that the peptizing effect would reach a maximum long before such high NaOH concentrations were reached, aside from the fact that there is a strong influence against hydrogen evolution unless the simple ionic equation is modified by the introduction of sodium hydroxide as a reactant.

There is another question that may be raised. The theory is that iron reacts with water to form a protective oxide film. This film covers the ferrite grains but perhaps is less adherent at the grain boundaries. NaOH peptizes this film to leave the metal free to corrode, but attack is concentrated at the grain boundary because stress concentration there makes them anodic to the ferrite. Is it necessary to assume reactions of iron compounds there according to Eq. 18? This is a point which to me, at least, is not clear and I should like to request a little more elaboration on the importance of compounds in the grain boundary attack.

Mr. Richard C. Corey[2] (*presented in written form*).—Mr. Tajc's explanation of the role of silica in promoting corrosion cracking by peptizing the iron oxides and subsequent protection of ferrite grains preferentially, thus allowing the stressed grain boundaries to fail without benefit of film protection, raises a question in my mind. Can we assume any film of this nature to be discontinuous or heterogeneous? Does it not seem highly improbable that the grain boundaries, of infinitely small area compared

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to the ferrite grains, should be unprotected by a precipitated oxide film? Assume for the moment that the oxide film was initially produced on the ferrite only. The action of caustic and silica produces a precipitate which by nature should distribute itself over the entire surface of the metal. Mr. Tajc's suggested mechanism may indeed present the true picture of the mechanism of corrosion cracking but the probability of certain factors remains open to question.

MR. J. A. TAJC[3] (*author's closure*).—Before answering the several questions, it seems necessary to explain the term "spontaneity" which has engendered considerable informal discussion. In chemical terminology, spontaneity is the tendency of substances to pass from one physical or chemical form to another; it is measured in terms of the free energy change or the entropy change of the system, and is not a measure of the reaction rate or velocity. The activity undoubtedly influences the reaction rate but it is far from being the determining factor.

Whether a very low hydrogen ion activity will influence the spontaneity of hydrogen evolution can be determined by formulating equations analogous to Eqs. 4 to 9, while using therein $Fe(OH)_2$ instead of Fe_3O_4, and substituting H^+ for OH^- in Eq. 4. The ultimate conclusion is "that the only way to decrease the spontaneity of the corroding reactions in water solutions is to exclude the water from contact with the iron." However, the rate of hydrogen evolution is in the domain of kinetics; so that, while the spontaneity of a reaction can be predicted from related thermodynamic data, the velocity of the same reaction cannot be even estimated. In general, experimental evidence similar to that of Fig. 1 is used to determine the reaction rate. The hydrogen ion activity does not appear in Eq. 9; consequently, there is no reason to believe that it should influence the rate of hydrogen evolution, except as it may alter the surface condition. This specific non-chemical accelerating action was assumed to be peptization. The validity of this has not been determined, and it is probable that some modification will be necessary as our knowledge of surface phenomena expands.[4, 5]

Since intercrystalline cracking is an essential feature of caustic embrittlement, the material of the grain boundary holds an important position in any explanation of the phenomena. Maier[6] has shown that the boundaries of a high purity metal have a greater density, higher energy content and are presumably the result of lattice deformation. The higher energy of the grain boundary would account for its greater susceptibility to etching and corrosion; so, the cracking may be explained without using metallic compounds as the corroding material in Eq. 18.

The belief that the protective film can be discontinuous at the grain boundary is supported by the large mass of evidence[5, 7, 8, 9] that all chemical reactions are atomically heterogeneous and obey statistical laws. The orientation of oil molecules on water, of gases on silica and charcoal; and anisotropy of all types are typical examples of heterogeneity. It is quite conceivable that a film could be discontinuous at boundaries more than one thousand atomic diameters wide. A more thorough discussion of the subject may be found in the texts previously mentioned.

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[3] Metallurgist, Power Stations Dept., Duquesne Light Co., Pittsburgh, Pa.

[4] R. F. Mehl, "Oxide Films on Iron," *Metals Technology, Technical Publication No. 780*, Vol. 4, No. 2 (1937).

[5] G. M. Schwab, H. S. Taylor and R. Spence, "Catalysis," pp. 7-19, 90-167, Chapters XI, XIII, XV, D. Van Nostrand Co., New York City (1937).

[6] Charles G. Maier, "Theory of Crystal Aggregates," *Metals Technology, Technical Publication No. 701*, Vol. 3, No. 3 (1936).

[7] Jerome Alexander, "Colloid Chemistry," D. Van Nostrand Co., New York City (1937).

[8] James William McBain, "The Sorption of Gases and Vapours by Solids," George Routledge and Sons, Ltd., London (1932).

[9] C. P. Smyth, "Dielectric Constant and Molecular Structure," Reinhold Publishing Corp., New York City (1931).

SOME APPLICATIONS OF THE POLARIZING MICROSCOPE TO WATER-CONDITIONING PROBLEMS

By Everett P. Partridge[1]

Synopsis

Chemical analysis of the highest precision is not adequate in some cases to indicate the actual substances deposited from water. As an adjunct to chemical analysis, the polarizing microscope may be used to identify the actual crystalline constituents of deposits by measurement of the refractive indices and other optical properties. At the same time the polarizing microscope is a useful tool for the identification of substances by chemical tests on small amounts of material, as well as for the general examination of all sorts of samples.

This paper presents a general discussion of the uses of the polarizing microscope in a laboratory devoted to water-conditioning problems, discussing in a general manner the technique employed in the examination of scale deposits and corroded metal surfaces, the application of chemical microscopy for the identification of substances, and the identification of crystalline materials by the measurement of optical properties. The purpose is not to present detailed directions for the use of the polarizing microscope, but to indicate the various ways in which it may be employed as a tool by a person not specifically trained in crystallography.

Much of the ancient art of water treatment is suggestive of alchemy and crystal gazing. In the developing science of water conditioning, however, alchemy has given way to physical chemistry and crystal gazing to the use of the polarizing microscope. Hall and Merwin[2] were the first to use this instrument broadly in the study of deposits from boiler waters. Later, Sulfrian[3], Hantel[4], and Powell[5] applied it to special problems in this same field. Only recently, however, has the polarizing microscope achieved its proper place in the study of water conditioning. To indicate briefly some of its uses is the purpose of this paper.

In the conditioning of water for industrial use, the chemist or the chemical engineer must repeatedly determine what is present in solution in water, what has been deposited from water, and how water has acted upon confining surfaces. The waters which he must examine are of infinite variety, as are the effects they produce in steam generators, pipe lines, heat exchangers, and process equipment in general.

In his efforts to predict the behavior of a water supply, or to determine the

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[1] Director of Research, Hall Laboratories, Inc., Pittsburgh, Pa.

[2] R. E. Hall and H. E. Merwin, "The Solid Phases Developed in Boiler Waters," *Transactions*, Am. Inst. Chemical Engrs., Vol. 16, Part II, p. 91 (1924).

[3] A. Sulfrian, "The Estimation of Steam Boiler Damage and Operation Period by Means of Optical Testing of the Deposits," *Wasser*, Vol. 5, p. 162 (1931).

[4] A. Hantel, "Microscopic Examination of Boiler Scale," *Die Wärme*, Vol. 54, p. 409 (1931).

[5] S. T. Powell, "A Critical Study of Boiler Scales and Advanced Methods of Analysis and Identification," *Combustion*, Vol. 5, No. 3, p. 15 (1933).

character of a solid deposit, or to interpret a case of corrosion, in examining numberless proprietary compounds or in identifying unexpected products of his own research, the water-conditioning expert commonly relies upon chemical analysis to guide his judgment. In many cases, however, the polarizing microscope may be used to advantage either to supplement or to supplant the work of the analytical chemist.

At its best, chemical analysis will yield precise values for the percentage of calcium oxide and carbon dioxide in a sample of feed-line scale, indicating unequivocally that the deposit is essentially calcium carbonate. But is it calcite or aragonite? Chemical analysis cannot give the answer, which may in some cases be worth knowing. And chemical analysis, even of the highest accuracy, may be of practically no use in the case of some complex boiler deposits. Against the ultimate precision of the analytical mode of attack must be set its chief disadvantages: it is time consuming; and it frequently does not indicate the actual substances present.

Where it can be applied at all, the polarizing microscope will usually yield not only definite evidence concerning the presence of some particular substance, but also a fair estimate of its amount relative to other constituents. In many cases, the time required to procure semi-quantitative data on a familiar type of material will be considerably less than for equivalent knowledge obtained by chemical analysis.

FIELDS OF APPLICATION FOR THE MICROSCOPE

A single good microscope of the polarizing type supplied with the necessary accessories makes possible many different types of work, including the following:

1. General examination of objects at magnifications from a few diameters upward.
2. Chemical microscopy—the identification of specific ions by the formation of crystals of characteristic appearance when reagents are added to a minute amount of an unknown material on a microscope slide.
3. Identification of crystalline substances by measurement of their indices of refraction and other optical properties.
4. The study of the behavior of material in the colloidal state of dispersion by means of dark-field illumination.
5. Measurement of particle size.
6. Determination of melting points and temperature-composition diagrams.
7. Metallographic observations.
8. Microprojection for demonstration and lecture purposes.
9. The recording of information in the form of photomicrographs.

In the present paper, discussion is confined to the first three of these ways in which the microscope may be used.

GENERAL EXAMINATION OF OBJECTS

A relatively slight magnification of an object will, in many cases, reveal structural details of considerable diagnostic significance. Obviously, the sample must be as nearly as possible in its original condition. While the chemist inevitably tends to grind up a sample so that he may determine its average composition, the most significant feature of a boiler deposit may be its variation in composition, which can only be determined by the microscopist if he has hand samples which have been disturbed as little as possible.

The chief requirement for general examination of opaque samples by reflected light is an objective with a good depth of focus so that as much as possible of the heights and valleys of an irregular surface may be seen at once. A 3.2× objective may be used to advantage with 5× and 10× eyepieces for such work. For surfaces which are relatively smooth, an objective as high as 10× may be used with these same eyepieces to give higher magnifications.

General examination at magnifications from 16 to 100 is frequently of great assistance in the study of boiler or feed-line deposits or of corroded specimens of metal. What to the eye alone appears simply as a series of indistinct bands on a cross-section of a piece of boiler scale may be seen under the microscope as layers of definitely different structure, and hence possibly of different composition. For example, layers of anhydrite scale can usually be recognized because of their typically coarser crystallization when they are sandwiched in between silicate layers in a complex scale.

The cross-section of a scale may sometimes be prepared to advantage by a treatment similar to that given specimens for metallographic examination. A smooth surface is produced by rubbing on sheets of progressively finer emery paper. This surface is then etched by immersion for a short time in a solution which will dissolve certain constituents of the scale more rapidly than others. For example, dilute hydrochloric acid will remove within 30 sec. any calcium carbonate or calcium phosphate in the polished surface layer, without affecting appreciably such constituents as calcium sulfate or most of the silicates. After the specimen has been rinsed in distilled water, dried in a jet of compressed air, and mounted edge up on a slide by means of plasticine, these more resistant constituents will appear in relief when the scale is examined with inclined illumination at a magnification of 100. For boiler scales, such a procedure yields as much information as the preparation of thin sections, and is much more simple and rapid. At most, however, it only indicates whether or not a scale is homogeneous and in some cases hints at the possible constituents.

In the examination of metal surfaces at low magnifications, the purpose of the water chemist is quite different from that of the metallographer. Where the latter is interested in grain size or the distribution of pearlite in a specially prepared highly-polished surface, the former is searching on a corrosion-roughened surface for the significant results of chemical action which will help him in reconstructing the process which has taken place. For this purpose, it is essential that the surface be as nearly as possible in the service condition. A well-meant cleaning-up by some workmen may render a specimen worthless.

Although chemical tests under the microscope are usually desirable to identify the constituents of proprietary compounds, preliminary general examination frequently will save time by revealing the presence of the typical forms of the usual industrial chemicals. With respect to the products of research, in a laboratory equipped with a polarizing microscope, it is the first tool to be used when an unexpected precipitate is encountered or a new product is being prepared. In many cases only a few minutes will suffice to indicate whether or not a material is a single substance or whether it is obviously a mixture.

IDENTIFICATION BY CHEMICAL TESTS

It is almost instinctive to turn from general examination of a sample to qualitative tests which may give a clue concerning its chief constituents. Many of the tests ordinarily used by the chemist for identification may be carried out to advantage under the microscope with only minute amounts of material. In addition, a large number of special tests for various metals and acid radicals have been developed.[6]

For the examination of deposits from water, dilute hydrochloric acid (1:1) is a most useful reagent. If its addition to a sample of scale from a feed line or a boiler produces an immediate fizz of bubbles, it is highly probable that calcium carbonate is an important constituent of the deposit, although sulfite or readily decomposed sulfides will produce the same effect. In addition to indicating the presence of salts such as carbonate or sulfite, hydrochloric acid dissolves calcium phosphate rapidly and completely, and slowly attacks many silicates, leaving a characteristic residue of hydrated silica.

Since the resistance of silicates to hydrochloric acid increases, in general, with the ratio of silica to metal oxide, it is usually possible to guess whether or not a particular constituent observed under the microscope while in contact with hydrochloric acid is high or low in silica.

The presence of copper in many deposits from boilers has recently excited a great deal of interest. The silver nitrate test suggested by Ward[7] may be used to determine whether copper indicated by chemical analysis is present as the metal or as a compound. A convenient procedure comprises stirring a minute portion of the powdered sample into a drop of distilled water on a slide, illuminating the drop from above at an angle, and observing one or more particles which show the typical red color and luster of copper while a drop of silver nitrate solution is added to the slide and brought in contact with the drop of water. Brightly reflecting silver "trees" will grow on the particles if they are actually metallic copper. Red particles of ferric oxide, which may sometimes be mistaken for copper by visual examination, do not, of course, respond to this test. Fragments of metallic iron, which are frequently present as small chips cut from the tube surfaces by turbine cleaners, also form the tree-like growths, but may be easily distinguished from metallic copper by their initial dark or silvery appearance.

When proprietary compounds are to be examined, a series of selected pH indicators prepared as solutions in 90 per cent alcohol may be used in much the same way that the biologist uses stains. Representative particles of the compound are spread upon a slide and a drop of indicator solution added. Because the inorganic salts are relatively insoluble in the 90 per cent alcohol, the indicator color is developed on or close to the particles. By testing with indicators it is thus possible to decide rapidly whether the phosphate in a compound is primary, secondary, or tertiary.

The indicator solutions are equally useful in checking the alkalinity of deposits from boilers. Although this may not be generally important, in cases where cracking has occurred in riveted seams it is desirable to determine whether or not traces of deposit from the inner surfaces of the seam or from the rivet holes are highly alkaline.

In passing, it might be remarked that the ancient problem of predicting what solid substances will be formed from a specified water when it is treated or evaporated in a prescribed manner may be solved by carrying out the desired process on a sample of the water, and then applying chemical microscopic methods to identify whatever solid phases have developed.

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[6] E. M. Chamot and C. W. Mason, "Handbook of Chemical Microscopy," Vol. 2, John Wiley and Sons, New York City (1931).

[7] C. J. Ward, "The Detection of Free Metal Particles in Dust," *The Analyst*, Vol. 58, p. 28, (1933).

IDENTIFICATION BY OPTICAL PROPERTIES

Aside from metallic materials of construction, most of the samples which interest the water chemist or engineer comprise substances which are fundamentally non-opaque when broken down into fine graments and observed under the microscope. If examined with an ordinary microscope, such particles generally yield few, it any, clues to their identity. By using polarized light, however, it is possible to measure one or more of a dozen optical properties for each of the constituent crystalline substances. Since no two substances have ever been found to display exactly the same combination of optical properties, identification of any substance by microscopic methods is at least theoretically possible.

Practically, the polarizing microscope has some definite limitations. Unless the individual particles have a major dimension of at least 10 μ, or, if smaller, have a characteristic crystal habit or cleavage, little can be done beyond determining the refractive indices. It is therefore difficult to identify without supporting chemical evidence the constituents of material in an extremely fine state of subdivision or in the form of microcrystalline aggregates from which individual particles larger than 10 μ cannot be isolated. Solid solutions present another difficulty, since the optical properties vary continuously from one end member to the other. Prior knowledge of the existence of such solid solutions is necessary for proper identification.

An important fundamental limitation in many cases, aside from those of solid solutions, is the lack of reference data for the substances to be identified. This necessitates the initial preparation of pure samples of the various substances whose occurrence is anticipated, and the careful correlation of their optical properties with their chemical composition before the polarizing microscope may be used as a tool for purposes of identification.

Although the further point might be raised that the microscope requires a special technique, this scarcely constitutes a limitation. Any person with normal vision and intelligence, patience, and a care for details, can develop in a few months the skill in manipulation and the judgment in interpretation which will allow him to use the polarizing microscope as a tool. While a knowledge of crystallography is, of course, a definite advantage, it is by no means a prerequisite for practical work; as a matter of fact, the person who makes use of the polarizing microscope will gradually teach himself a creditable amount about crystallography.

Since detailed discussions of the polarizing microscope and of the technique of its use are available[8, 9] only a brief outline is justified here to show the procedure in the study of a solid sample. To begin with, after general examination of a piece of the sample at low magnification, a fragment about $\frac{1}{32}$ in. in each dimension is broken off from any chosen definite layer or zone, and is crushed to a powder on a slide with as little grinding as possible. About one-tenth of the resulting fine powder is transferred to a second slide, a drop of a standard liquid with a known refractive index is allowed to fall on it, and a cover glass is placed on the mixture. The material is then examined under the microscope, using both polarized light and ordinary illumination. In working with the small particles which constitute most deposits from water, the author has come to use a 45× objective with a 10× eyepiece as a standard

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[8] N. H. Hartshorne and A. Stuart, "Crystals and the Polarizing Microscope," Edward Arnold and Co., London (1934).

[9] E. M. Chamot and C. W. Mason, "Handbook of Chemical Microscopy," Vol. 1, John Wiley and Sons, New York City (1931).

arrangement. According to Saylor[10] the use of an objective of lower power, such as the 20×, leads to more accurate measurement of the refractive indices. In the work under consideration, however, the increased visibility of small particles afforded by the 45× objective more than offsets any small loss in precision. Even with the 45× objective, routine measurements of index should be accurate to ±0.003, which is quite adequate for purposes of identification.

Examination of the first slide prepared from a sample should yield two important bits of information. In the first place, certain distinguishing characteristics of the particles may suggest their identity. For example, the natural cleavage of anhydrite ($CaSO_4$) when it is crushed tends to produce fragments with square corners and parallel extinction, while minute particles of calcite ($CaCO_3$) show up in polarized light like brilliantly colored stars, due to the unusually high birefringence of this substance. In the second place, the initial examination should indicate whether the sample contains essentially one, two or more major crystalline constituents. Judgment in this matter is based upon typical differences in appearance, relative differences in refractive index between the particles and the liquid in which they are immersed, and the birefringence, extinction position and sign of elongation of the particles. Where the individual particles are sufficiently large, further distinction may be made on the basis of interference figures.

After the number of constituents has been estimated and possibly the identity of one or more has been guessed from observation of the first slide, one of the constituents is chosen for further study and designated as *A*. By comparison of the refractive indices of this constituent with immersion liquids of known index on successive slides, the minimum and the maximum indices may be determined.

At the same time, the indices of the other constituents, *B*, etc., may be estimated roughly, and when favorably oriented particles of sufficient size are found, interference figures may be studied to determine whether a constituent is uniaxial or biaxial and positive or negative.

In measuring refractive indices, the standard Becke bright-line method employing central illumination is the most convenient to use and is sufficiently precise for all ordinary purposes. Saylor[10] has developed a double-diaphragm inclined-illumination method, which is said to improve the precision in determining the match between the index of the substance under observation and the liquid in which it is immersed.

In the case of isotropic substances, such as sodium chloride or analcite, the index of refraction is the same in all possible directions through each crystal, so that a measurement may be made upon any particle regardless of its orientation. In the case of uniaxial or biaxial substances, however, the refractive index differs with the orientation of the particle. To determine the principal indices, two for uniaxial, and three for biaxial crystals, the procedure recommended by Slawson and Peck[11]—is preferable when the particles are large enough to allow the observation of interference figures. In most samples of deposits from industrial waters, however, the particles are so small that it is only possible to apply the statistical method of finding the highest and lowest indices

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[10] C. P. Saylor, "Accuracy of Microscopical Methods for Determining Refractive Index by Immersion." National Bureau of Standards *Journal of Research*, Vol. 15, p. 277 (1935).

[11] C. B. Slawson and A. B. Peck, "The Determination of the Refractive Indices of Minerals by the Immersion Method," *American Mineralogist*, Vol. 21, p. 523 (1936).

exhibited by particles of a given constituent. In general, it is possible to approximate these quite closely by observations on those particles showing the highest interference colors and hence the greatest difference in index when oriented in their respective extinction positions in polarized light.

Various other properties may be determined depending upon the character of the material under investigation and the training of the observer. The refractive indices, however, are always the most important and frequently the only properties measurable in the identification of a substance.

After the optical properties of constituent *A* have been determined in so far as the character of the sample permits the procedure is repeated until the properties of the other constituents have been measured. Depending upon the complexity of the sample and the precision of the information desired, from ten minutes to a day of study may be required to work out the composition of the small fragment originally removed. If the deposit consists of layers or zones of apparently different composition, the process must then be repeated for each layer or zone. The final step in identification is the comparison of the observed data with reference information already available in published form or measurements made on special samples of known composition.

By estimating the relative number and size of particles of the various constituents in several different fields under the microscope, a semi-quantitative analysis may be made for the chief constituents. Thus, it is easy to determine whether one deposit is 50, 75, or nearly 100 per cent anhydrite, or whether another contains more calcite than calcium silicate.

A word should be said concerning contamination of samples. To the uninitiated, little spheres of fly ash or flakes of aluminum paint or fibers from cloth or paper may be the source of much bewilderment. The microscopist must develop a mild but unremitting skepticism concerning anything unusual he observes, not granting it a place as a true constituent of the sample until the evidence is fairly definite, but not, on the other hand, forgetting its presence.

SUGGESTED EQUIPMENT

To carry out the types of investigation with the polarizing microscope mentioned in this paper, an investment of several hundred dollars will be necessary. Individual preferences and budgets will cause the list of equipment to vary considerably. The following list merely represents one working combination:

Microscope.—A wide-field instrument with a well-built rotating stage, with an adjustable substage unit carrying the polarizer, condensing system, and iris diaphragm, and with the analyzer and Bertrand lens mounted on slides in the tube is preferable to the so-called "chemical microscopes," although competent work may be done with the latter. Provision for easy and accurate centering of the optical system with respect to the stage is important. A mechanical stage for convenient manipulation of the slide will more than pay for itself in time saved.

Optical System and Accessories.—Three objectives, 3.2×, 10× and 45×, combined with two eyepieces, 5× and 10×, with cross-hairs, will give a convenient combination of magnifications ranging from 16 to 450 diameters. For projection or photomicrographic work, a special eyepiece corrected to give a flat field is desirable. Any one of several types of micrometer eyepiece when properly calibrated, will make possible quantitative measurements of scale thickness or particle size.

A gypsum plate is a generally useful accessory. For the type of work described in this paper, the standard mica plate and quartz wedge are less important.

Illumination.—A microscope lamp equipped with a concentrated-filament bulb and a support for glass filters will be suitable for all purposes except microprojection or photography at very high magnifications. For these latter types of work an arc-lamp is required.

Refractive Index Standards.—A set of refractive index liquids covering the range from 1.36 to 1.76 is desirable, although for special purposes it may be necessary to have available only a limited number of standard liquids corresponding to the principal indices of certain substances. The author employs a set arranged in steps of 0.010 from 1.360 to 1.470, and from 1.660 to 1.760, and in steps of 0.005 in the intermediate range from 1.470 to 1.660. Such a set may be purchased ready for use from various supply houses or individuals, or may be prepared by the user provided he has access to a refractometer.

Reagents for Chemical Microscopy.—Standard sets of the reagents suggested by Chamot and Mason[9] may be obtained from supply houses. In many cases, however, a limited number of reagents in ordinary dropper bottles will suffice.

Slides and Cover Glasses.—For use with a mechanical stage, slides 1 by 2 in. are much to be preferred to longer slides. Cover glasses $\frac{1}{2}$ in. square of No. 2 thickness are convenient to use.

Implements.—For handling small particles or minute quantities of powdered sample, the dissecting tweezers, probe, and section lifter commonly used by zoology students are ideal. In carrying out chemical tests, small glass rods may be shaped as desired for the addition of reagents to a slide. Chamot and Mason[9] describe various convenient devices for special operations in chemical microscopy.

Reference Data.—The most convenient and least expensive source of data for the natural minerals is *Bulletin 848* of the United States Geological Survey.[12] This should be supplemented by the comprehensive collection of data for artificial minerals made by Winchell.[13] New information appearing from time to time may be found readily by reference to *Chemical Abstracts* under the headings of *refractive index*, *optical properties*, *microscopy* and *petrography*.

PROBLEMS SOLVED AND UNSOLVED

No statement in this paper should be construed to indicate that the polarizing microscope will yield an answer to every problem. In many cases, however, it will supply essential information obtainable in no other way. Much work must still be done before it will be possible to present a reasonably complete scheme for the examination of deposits from water. Some progress has been made, however, in the past year. In addition to the easily identified three forms of calcium sulfate, two forms of calcium carbonate, and single forms each of calcium hydroxide and magnesium hydroxide previously tabulated,[14] of which anhydrite ($CaSO_4$) and calcite ($CaCO_3$) are frequently observed, definite identification has been made of the following: quartz (SiO_2), halite ($NaCl$), and thenardite (Na_2SO_4) in turbine-blade deposits; quartz and analcite ($Na_2O \cdot Al_2O_3 \cdot 4SiO_2 \cdot$

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[12] E. S. Larsen and H. Berman, "The Microscopic Determination of the Nonopaque Minerals," *Bulletin 848*, U. S. Geological Survey (1934).

[13] A. N. Winchell, "The Microscopic Characters of Artificial Inorganic Substances or Artificial Minerals," Seond Edition, John Wiley and Sons, New York City (1931).

[14] E. P. Partridge, "Formation and Properties of Boiler Scale," *Bulletin No. 15*, University of Michigan, Engineering Research, p. 41 (1930).

$2H_2O$) in scales from water-wall tubes in boilers; radiophyllite ($CaO \cdot SiO_2 \cdot H_2O$) in a boiler deposit; metallic copper in sludges and scales from a large number of boilers; elemental sulfur in a scale from the heater of a sulfite pulp digester; and calcium sulfite in a deposit from a chemical feed pump. Tricalcium phosphate has been found consistently as particles so small as to be barely resolved. In a few cases constituents believed to be dahlite, $2Ca_3(PO_4)_2 \cdot CaCO_3$, or $Ca_3(PO_4)_2 \cdot Ca(OH)_2$ have been observed.

Perhaps the greatest problem still unsolved in relation to deposits from water is the question of the constitution and mode of formation of silicate scales. Here the polarizing microscope may play an important part in conjunction with other methods of investigation.

DETERMINATION OF HARDNESS IN WATER BY DIRECT TITRATION

BY R. T. SHEEN[1] AND C. A. NOLL[1]

SYNOPSIS

Hardness in water may be determined by a direct titration with the potassium salt of certain fatty acids. Potassium stearate and potassium palmitate are found to be of value, the palmitate allowing the more accurate determination in the presence of interfering ions. Comparisons are given with the standard soap method on a number of samples, the results showing the palmitate method to be subject to less error. The more extensive use of the palmitate method is suggested.

Hardness in water is now determined in most laboratories by a measurement of the amount of soap solution required to give a permanent lather. This method gives a rough approximation of the amount of calcium and magnesium present, and might be referred to as a measurement of the "foaming capacity" **(5)**[2] of the water. Reference has been made to the fact that differences between hardness as found by soap and as calculated from complete analysis occur because calcium and magnesium salts do not react with soap in equivalent proportions **(6)**.

Hydrolysis methods employing the potassium salts of some fatty acids were first suggested by Blacher **(1)**, using potassium stearate, and later potassium palmitate **(2)**. In his method, hardness is determined by titration using methyl orange as an indicator following exact neutralization and boiling to liberate carbon dioxide. Froboese **(3)** stated that a separate determination of magnesium could be made in the presence of calcium by precipitation of the calcium as calcium oxalate and titration of the magnesium by potassium palmitate in the presence of the calcium oxalate precipitate. Duroudier **(4)** separates the calcium oxalate by filtration before titration of the magnesium and further claims a sensitivity of 1.4 mg. CaO or 1 mg. MgO per liter, using potassium palmitate.

Duroudier prepared his standard potassium palmitate with propyl alcohol, which he found better for this purpose than ethyl alcohol. Hamer **(7)** claims an accuracy in determination of total hardness by potassium palmitate of 5 p.p.m. and discusses the possible use of this titration in the determination of sulfate. Bond **(8)** discusses a comparison of the palmitate method and results with the results obtained by the Hehner method, soap solution and calculation from gravimetric analysis. He concludes on the basis of comparison of results on five different types of water that the potassium palmitate method gives results for calcium and magnesium more accurate than can be obtained by either of the other methods.

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[1] Technical Director, and Chief Chemist, respectively, W. H. & L. D. Betz, Philadelphia, Pa.

[2] The boldface numbers in parentheses refer to the reports and papers given in the list of references appended to this paper, see p. 613.

The main purpose of this paper is to check the possible application of the palmitate method to routine analysis for hardness, and to present the accumulated evidence in favor of a more general adoption of this method. In this study, a check was made on the possible advantage of using potassium stearate, oleate or laurate. No endpoint was obtained with potassium laurate. Potassium oleate gave an endpoint value in low titrations, but was not applicable over any appreciable range. Data were obtained in detail on both potassium palmitate and potassium stearate, although results clearly indicate the superiority of the palmitate.

PREPARATION OF REAGENTS

Special Solutions:

Potassium palmitate, 1 ml. = 1 mg. $CaCO_3$ (0.02 *N*): To make 5 l. standard potassium palmitate solution, mix 4500 ml. ethyl alcohol (80 per cent) (formula No. 30 or No. 3-A may be used) with 500 ml. c.p. glycerine. To 500 ml. of this mixture, add 25.6 g. of palmitic acid and 1 ml. of a 1 per cent solution of phenolphthalein. Heat on a water bath until solution is complete. Dissolve 6 g. of c.p. potassium hydroxide in 100 ml. of the remaining alcohol glycerine mixture and add this solution dropwise from a burette to the palmitic acid solution until a permanent pink color is obtained. Dilute the resulting solution of potassium palmitate to 5 l. with the remaining alcohol and glycerine mix, using 80 per cent alcohol for final volume correction. The added alcohol-glycerin should restore the solution to colorless. Should the solution remain pink, add 0.05 *N* hydrochloric acid dropwise until the pink color is just discharged.

Potassium Stearate: Use the same procedure as described for preparation of potassium palmitate, substituting 28.5 g. of stearic acid for the 25.6 g. of palmitic acid.

Standard Solutions:

Hydrochloric acid, 0.05 *N*.

Sodium hydroxide, 0.05 *N*.

Phenolphthalein, 1 per cent solution.

Calcium nitrate $Ca(NO_3)_2$, 1 ml. = 1 mg. $CaCO_3$: Dissolve 1.641 g. calcium nitrate to 1 liter of solution and standardize by the usual potassium permanganate titration method.

STANDARDIZATION OF POTASSIUM PALMITATE

Pipette 10 ml. of the calcium nitrate solution and dilute to 50 ml. and place sample in a No. 5 evaporating dish. The sample should be well illuminated for titration. Add 0.5 ml. of phenolphthalein indicator and adjust the solution just to the acid side of phenolphthalein. Titrate with potassium palmitate to a definite pink end point, stirring the sample vigorously during the titration. Three checking values should be obtained. The blank determination should be made on a 50-ml. sample of distilled water adjusted to phenolphthalein end point with 0.05 *N* sodium hydroxide and 0.05 *N* hydrochloric acid and titrated to a definite pink with potassium palmitate. This value should be subtracted from the regular titration before calculation of results and with approximately 0.02 *N* potassium palmitate will be found to be about 0.3 ml.

The standards used in this investigation had the following values:

Potassium palmitate, 1 ml. = 0.983 mg. $CaCO_3$
Calcium nitrate, 1 ml. = 0.975 mg. $CaCO_3$
Magnesium chloride, standardized gravimetrically as $Mg_2P_2O_7$, 1 ml. = 1.096 mg. $CaCO_3$
Magnesium sulfate, standardized gravimetrically as $Mg_2P_2O_7$, 1 ml. = 0.673 mg. $CaCO_3$

TEST PROCEDURE

A 50-ml. sample of water is measured into a No. 5 evaporating dish and 0.5 ml. of phenolphthalein indicator is added. If the sample is alkaline to phenolphthalein, neutralize just to a colorless end point. If the sample is originally acid to phenolphthalein, titrate to the phenolphthalein end point with 0.05 *N* sodium hydroxide and just discharge the red color with 0.05 *N* hydrochloric acid.

TABLE I.—DIRECT TITRATION OF STANDARD SOLUTIONS OF CALCIUM AND MAGNESIUM WITH POTASSIUM PALMITATE AND POTASSIUM STEARATE.

Reagent	Present		Found		Error	
	mg.	p.p.m.	mg.	p.p.m.	mg.	p.p.m.
Potassium Palmitate, ml.[c]	CALCIUM AS $CaCO_3$					
0.90	0.5	10	0.6	12	0.1	+2
1.40	1.0	20	1.1	22	0.1	+2
2.40	2.0	40	2.1	42	0.1	+2
5.30	5.0	100	5.0	100	0.0	0
10.10	9.8	196	9.6	193	0.2	−3
19.90	19.6	392	19.3	386	0.3	−6
	MAGNESIUM AS $CaCO_3$					
1.40[a]	1.1	22	1.1	22	0.0	0
5.60[a]	5.5	110	5.2	104	0.3	−6
10.95[a]	11.0	220	10.5	210	0.5	−10
2.95[b]	2.7	54	2.6	52	0.1	−2
12.10[b]	12.1	242	11.6	232	0.5	−10
Potassium Stearate, ml.[d]	CALCIUM AS $CaCO_3$					
0.60	0.5	10	0.3	6	0.2	−4
1.00	1.0	20	0.7	14	0.3	−6
2.05	2.0	40	1.8	35	0.2	−5
19.90	19.6	392	19.6	392	0.0	0
	MAGNESIUM AS $CaCO_3$					
1.25[a]	1.1	22	1.0	19	0.2	−3
5.00[a]	5.5	110	4.7	94	0.8	−16
21.60[b]	21.9	438	21.3	426	0.6	−12
0.60[b]	0.7	14	0.3	6	0.4	−8
4.90[b]	5.4	108	4.6	92	0.8	−16

[a] Mg Cl_2.
[b] Mg SO_4.
[c] 1 ml. potassium palmitate = 0.983 mg. $CaCO_3$ = 19.7 p.p.m. $CaCO_3$ (50-ml. sample).
[d] 1 ml. potassium stearate = 1.00 mg. as $CaCO_3$ = 20 p.p.m. as $CaCO_3$.

Titrate with 0.02 *N* potassium palmitate, stirring vigorously until a permanent red color is developed that will not disappear upon stirring for 30 sec.

CALCULATION OF RESULTS

From the number of milliliters of potassium palmitate required for the titration, subtract 0.3 ml. for a blank and multiply the results by 20 to obtain the hardness of the sample in parts per million according to the following equation: in (milliliters potassium palmitate $-0.3) \times 20 =$ p.p.m. hardness as $CaCO_3$ when a 50 ml. sample is taken for titration.

TABLE II.—EFFECTS OF VARYING CONCENTRATIONS OF IONS ON END POINT IN THE PALMITATE METHOD.

Potassium Palmitate, ml.[a]	Ion Present, p.p.m.	Calcium as $CaCO_3$, p.p.m.		Magnesium as $CaCO_3$, p.p.m.		Error, p.p.m.
		Present	Found	Present	Found	
	SiO_2					
0.75....	10	10	9			−1
10.00....	10	195	191			−4
10.00....	25	195	191			−4
2.30....	10			44	39	−5
3.20....	25			44	57	+13
	SO_4 as Na_2SO_4					
0.90....	800	10	12			−2
9.90....	800	195	189			−6
1.30....	2000	10	20			+10
9.80....	2000	195	187			−8
9.65....	4000	195	184			−11
2.50....	800			44	43	−1
2.60....	2000			44	45	+1
	(Fe)+++					
0.70....	5	10	8			−2
0.90....	10	10	12			+2
	Cl as NaCl					
0.85....	200	10	11			+1
0.80....	2000	10	10			0
9.80....	2000	195	187			−8
2.85....	200			44	50	+6
1.90....	2000			44	32	−12

[a] 1 ml. potassium palmitate = 0.983 mg. $CaCO_3$ = 19.7 p.p.m. $CaCO_3$.

EXPERIMENTAL RESULTS AND EFFECTS OF IONS

Table I gives the results of titration of standard calcium nitrate, magnesium chloride and magnesium sulfate with potassium palmitate and potassium stearate. With potassium palmitate, calcium can be titrated to a concentration as high as 400 p.p.m. as calcium carbonate, but the magnesium cannot exceed 200 p.p.m. as calcium carbonate and still obtain a sharp end point. With potassium stearate, results compare favorably with potassium palmitate in titrating calcium, but the titration of magnesium gives erratic results.

TABLE III.—RESULTS OF TOTAL HARDNESS DETERMINATIONS ON VARIOUS SAMPLES, WITH COMPARISONS AGAINST GRAVIMETRIC ANALYSIS.

Sample	Calcium as $CaCO_3$ Gravimetric, p.p.m.	Magnesium as $CaCO_3$ Gravimetric, p.p.m.	Calculated Total Hardness as $CaCO_3$, p.p.m.	Hardness with Potassium Palmitate, p.p.m.	Hardness with Potassium Stearate, p.p.m.	Hardness, Clark Method, p.p.m.	
						Neutralized	Not Neutralized
No. 1.....	23.3	10.2	33.5	27	26	27	30
No. 2.....	20.6	10.1	30.7	27	18	26	43
No. 3.....	23.9	17.9	41.8	43	40	24	34
No. 4.....	45.2	10.2	55.4	44	42	51	54
No. 5.....	9.3	2.5	11.8	12	6	11	16
No. 6.....	21.3	8.6	29.9	27	26	31	36
No. 7[a]....	683	349	1032	1000	824	910	945
No. 8[a]....	1001	495	1496	1490	1276	1340	1250

[a] Dilution factor of 5 used in all tests.

TABLE IV.—ANALYSIS OF WATER SAMPLES.

Sample	SiO_2 p.p.m.	PO_4 p.p.m.	Fe^{+++} p.p.m.	$SO_4^{=}$ p.p.m.	Cl^- p.p.m.	$(HCO_3)^-$ as $CaCO_3$ p.p.m.	$(CO_3)^=$ as $CaCO_3$ p.p.m.	$(OH)^-$ as $CaCO_3$ p.p.m.	pH
No. 1...	3.5	0	0.1	34	2	88	0	0	6.9
No. 2...	3.0	0	0.1	6	1	34	0	0	5.9
No. 3...	18.5	28	0.0	448	104	200	160	0	10.5
No. 4...	12.3	0	0.1	496	136	328	32	0	8.5
No. 5...	0.0	0	0.1	2	0	12	0	0	6.1
No. 6...	2.5	0	0.1	32	2	36	0	0	6.7
No. 7...	2.0	0	5.0	496	9	288	0	0	7.1
No. 8...	2.0	0	5.1	514	11	298	0	0	6.9

The effects of various ions commonly found in water were investigated and results are shown in Table II. Silica, introduced in the form of sodium silicate, shows some interference with magnesium in higher concentrations, although it is possible that limiting solubility of magnesium in such a system influences these results. The sulfate ion, when present in high concentrations, approximately 2000 p.p.m. as Na_2SO_4, offers some interference with hardness in low concentrations but in all normal systems will not interfere. The ferric ion may be present up to 10 p.p.m. without appreciable interference. Chloride concentrations may be as high as 2000 p.p.m. as NaCl without material interference in calcium determination but show a slight interference in the determination of magnesium.

The method was applied to the determination of total hardness on a number of actual water samples and these data are shown in Table III. Results are given for comparison on determination with potassium stearate on some samples, and with Clark's method on some samples not neutralized and also neutralized to pH 8.3 before titration. Gravimetric calcium and magnesium was determined in each case. Table IV shows a regular mineral analysis of these samples.

DISCUSSION AND CONCLUSION

A comparison of the values obtained in the palmitate method with the gravimetric determination of calcium and magnesium in general shows good agreement, with an average variation in normal concentrations of 3 to 6 p.p.m. with an occasional value showing some discrepancy. Results are slightly better than those obtained with the Clark method on neutralized samples. The neutralization of a sample before titration with Clark's soap method seems to offer some improvement in that method, particularly in lower concentrations of hardness.

Time did not permit a study of the methods suggested by several previous investigators (**3**, **4**, **7**, and **8**) on a separate determination of magnesium following an oxalate precipitation of calcium, but this will be made the subject of further investigation.

Acknowledgment.—The comments and

suggestions of H. L. Kahler and other members of the Betz technical staff are appreciated. The authors gratefully acknowledge the generous aid of W. H. & L. D. Betz and their kind permission to present these data.

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DISCUSSION

Mr. T. H. Daugherty[1] (*presented in written form*).—One of the most interesting conclusions to be drawn from this paper is that the Blacher palmitate method yields results no more accurate than those obtained by the Clark method when the latter is carried out with proper regard for the fundamental properties of soap. About six years ago Kean and Gustafson[2] suggested that the erratic results of soap hardness tests on waters high in carbon dioxide were due to hydrolysis of the soap, and suggested the addition of sodium hydroxide to the standard soap solution. Several years ago in our own laboratory the standard procedure was adopted of adjusting each sample of water with dilute alkali or acid until it showed a faint pink color with phenolphthalein before addition of soap. This corresponds closely to the pH of 8.3 used by Sheen and Noll.

Adjustment of the sample in this manner serves to make the results on different samples at various initial pH values more strictly comparable with one another and with the calcium or magnesium solution used in standardizing the soap reagent. This procedure satisfactorily eliminates the interferences due to CO_2 and iron salts. Color of the sample does not influence the end point obtained. A blank value is obtained which is more constant and more easily reproducible than the lather factor obtained without this adjustment. It is noticed in Table III of the paper that the data obtained by such a neutralization step in the Clark method are in as good agreement with the gravimetric data as the data obtained with the potassium palmitate titration. Where the sample contains CO_2, iron salts or coloring agents, it is likely that the results by the adjusted Clark procedure will be more reliable, due to the interference of these materials in the color-change end point of the Blacher method as discussed by Kolthoff and Furman.[3]

Another improvement on the Clark procedure is the use of 80 per cent isopropanol for preparing the stock and reagent solutions. Precipitation and consequent change in standardization value, due to aging and to temperature changes, are materially reduced from those with the usual ethanol or methanol solvents.

Messrs. R. T. Sheen[4] **and C. A. Noll**[4] (*authors' closure*).—The purposes of our investigation was to see whether we could find a more accurate method and at the same time a method that would result in a saving in time. An examination of Table III will show an average deviation of 4.2 p.p.m. hardness on the titration with potassium palmitate as against a deviation of 5.9 p.p.m. with the use of the Clark method on a neutralized sample. Such an increase in the accuracy to be obtained is desirable. In our laboratories, analyses are made on from 75 to 300 samples each day. If we can reduce the time of an analysis by even 1 min. without sacrificing accuracy, we have effected a desirable change and for this reason have adopted the palmitate method for determination of hardness in practically all except boiler water samples. In boiler water and colored waters, we use the Clark method, neutralizing the sample to the phenolphthalein end point prior to titration.

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[1] Research Chemist, Hall Laboratories, Inc., Pittsburgh, Pa.

[2] R. H. Kean and H. Gustafson, "Some Factors Influencing Soap Tests for Hardness," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 3, p. 355 (1931).

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[4] Technical Director, and Chief Chemist respectively, W. H. & L. D. Betz, Philadelphia, Pa.

TECHNIQUE IN THE DETERMINATION OF DISSOLVED OXYGEN

By T. H. Daugherty[1]

Synopsis

A discussion is presented of the need for determining extremely small concentrations of dissolved oxygen in plant waters, particularly those pertaining to boiler plants. The various modifications of the basic Winkler procedure appearing in the literature are considered from the standpoints of sensitivity, accuracy and practicability in the plant. The Schwartz and Gurney double titration B modification, with several recommended changes in procedure, is believed to be the simplest to operate with a sensitivity and accuracy equal to other methods and hence most satisfactory. A detailed discussion of the theory and procedure of this modification is presented. Emphasis is placed throughout upon the careful technique that is essential in the determination of traces of dissolved oxygen. Recommendations are made for comparative tests of all methods, for further investigation of the electrometric titration and for determining the permissible limit in the sample of contaminants such as nitrite and sulfite.

The internal corrosion of metal in boilers and related equipment is a problem of ever-increasing importance to industry. Ever since dissolved oxygen was recognized as a primary factor in promoting corrosion, a continuous battle has been waged to limit the amount of it entering a boiler system. More recently, chemical treatment to remove residual traces of oxygen has been widely employed.

As a result of the wide use of mechanical deaeration to remove most of the dissolved oxygen from boiler feed water, methods of determining the residual oxygen content have received much attention. Improvement in the design and operation of deaerating equipment has inspired many efforts to increase the sensitivity and accuracy of the analytical methods as the quantities to be measured became steadily smaller.

The Winkler method is the basic test, particularly for field control use. It is generally considered to have a lower limit of determinability of 0.03 p. p. m. O_2 and a reproducibility of 0.01 p. p. m. O_2.[2] For many uses, these limits are sufficient, particularly where simplicity is a factor. However there have been cases where definite oxygen corrosion has occurred from waters consistently showing zero oxygen by this test. Thus it can readily be seen that the need for a test for dissolved oxygen, more sensitive and more accurate than the Winkler test, is imperative.

In the various tests that have been proposed in the literature to meet this need, three principles have been employed to eliminate or reduce the errors attributable to the Winkler procedure. These are: (1) evolution of the dissolved oxygen, so that it can be fixed and

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[1] Research Chemist, Hall Laboratories Inc., Pittsburgh, Pa.

[2] One part per million O_2 is equal to 0.7 ml. O_2 per liter at OC. and 760-mm. pressure.

titrated away from contaminants; (2) separate determination of the magnitude of the errors present, so that they can be eliminated by the use of factors; and (3) double titration, so that any errors present can be eliminated by subtraction of two titration values that are identical except for the constituent sought. It will be noted that all of these principles apply to modifications of the Winkler method. Other principles have been presented, some not based upon the Winkler method, but those listed above are the only ones claiming a greater sensitivity than the Winkler test.

All of these modifications require the use of an accurate technique commensurate with the sensitivity and accuracy expected. This is true of any test measuring concentrations in thousandths of a part per million, but from what has been observed in field use, the necessity for refinement of technique seems to be generally overlooked in connection with the oxygen test.

The procedure of White, Leland and Button[2a] employs the evolution principle. This method has not yet been reduced to a simplicity that would enable its general use in the field. Basically it still contains errors, such as oxygen and contamination in reagents and interference of volatile contaminants, that are incident to the Winkler test and that must be cancelled out by subtraction of predetermined factors.

The second principle of determining the magnitude of the individual errors has been utilized by Subcommittee VIII on Standardization of Water Analysis Methods of the Joint Research Committee on Boiler Feedwater Studies.[3] The accuracy obtained is predicated upon the accuracy in obtaining constants for the individual errors comprising the total recognizable error, and upon the constancy of these individual errors.

The procedures of Schwartz and Gurney[4] employ the third principle of double titration. It is believed by the author that the chances for accuracy are improved by the use of this principle because the aggregate of the individual positive or negative errors is cancelled out in each determination, and hence there is no need for dependence upon evaluation or constancy of the individual errors. Also the same steps in technique that can be applied for minimizing overall error, such as more dilute titrating reagent, electrometric titration without starch, etc., can be applied with equal benefits to a method based upon this principle as to one based upon either of the other two principles. Hence these methods will be discussed in detail.

The Schwartz and Gurney methods have received only a limited acceptance by those interested in testing for traces of dissolved oxygen, because of a general belief that the procedures are complicated and require too exacting a technique and because of the frequent lack of understanding of the mechanism by which errors normally incident to the Winkler test are cancelled out. Others qualified in the technique and thoroughly familiar with the theory have tried to follow the procedures, but have obtained unsatisfactory and inconsistent results, due, in the author's opinion, to faults in the procedures themselves which can be satisfactorily eliminated by the use of certain logical modifications.

It is proposed to present the A and B methods of Schwartz and Gurney in detail, discuss their theoretical back-

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[2a] A. H. White, C. H. Leland and D. W. Button, "Determination of Dissolved Oxygen in Boiler Feed Water," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 697 (1936).

[3] C. H. Fellows, "Determination of Dissolved Oxygen in Boiler Feedwater," Progress Report to Joint Research Committee on Boiler Feedwater Studies, May, 1935, not published.

[4] M. C. Schwartz and W. B. Gurney, "The Determination of Traces of Dissolved Oxygen by the Winkler Method," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 796 (1934).

ground, point out several sources of eorror, describe changes in technique to eliminate such errors and then present an adaptation of the B modification which is simple and foolproof so far as consistent with the accuracy and sensitivity required.

THEORY OF METHOD A

In the authors' closure to the discussion of their paper,[5] Schwartz and Gurney give the following outline of the procedure for method A:

> Four samples of which two [500-ml. Erlenmeyer flasks in tandem, containing three-holed rubber stoppers for an inlet and outlet tube as well as a solid glass plug for reagent addition] are collected in the usual manner and two of which may be collected in the open (are taken). Then add the following reagents in the order and quantities listed below shaking well after each addition of reagent:
>
> | SAMPLE T_1 | SAMPLE T_2 |
> |---|---|
> | 1 ml. $MnCl_2$ | 2 ml. $MnCl_2$ |
> | 1 ml. alk. KI | 2 ml. alk. KI |
> | 1 ml. H_2SO_4 | 2 ml. H_2SO_4 |
> | 1 ml. $KH(IO_3)_2$ | 1 ml. $KH(IO_3)_2$ |
>
> | SAMPLE B_1 | SAMPLE B_2 |
> |---|---|
> | 1 ml. alk. KI | 2 ml. alk. KI |
> | 1 ml. H_2SO_4 | 2 ml. H_2SO_4 |
> | 1 ml. $MnCl_2$ | 2 ml. $MnCl_2$ |
> | 1 ml. $KH(IO_3)_2$ | 1 ml. $KH(IO_3)_2$ |
>
> Take 500 ml. for titration with 0.01 *N* sodium thiosulfate.
>
> Then $2(T_1 - B_1) - (T_2 - B_2) =$ milliliters of oxygen in sample or for 500-ml. samples. [Note: This should read: Then $2(T_1 - B_1) - (T_2 - B_2) =$ milliliters of 0.01 *N* sodium thiosulfate equivalent to dissolved oxygen in 500-ml. sample.]
>
> $0.112 \times [2(T_1 - B_1) - (T_2 - B_2)] =$ milliliters of oxygen per liter in sample.

The titration values *T* and *B* throughout this paper are in terms of milliliters of 0.01 *N* sodium thiosulfate.

The titration value of sample T_1, in which 1 ml. of each reagent is added in normal Winkler order, is due to the summation of the iodine liberated or absorbed by the following factors:

1. Oxygen fixed from 500-ml. sample.
2. Oxygen fixed from 1 ml. of each of the first two reagents.
3. 1 ml. $KH(IO_3)_2$ reagent.
4. Starch end point error.
5. Interfering substances from 500-ml. sample.
6. Contamination other than dissolved oxygen from 1 ml. of each added reagent.

The titration value of blank B_1, in which 1 ml. of each reagent is added in reverse Winkler order, thus fixing no oxygen, is due to the summation of the iodine liberated or absorbed by the above factors 3, 4, 5 and 6.

Thus by subtracting:

$T_1 - B_1 =$ titration value of oxygen fixed from 500-ml. sample + titration value of oxygen fixed from 1 ml. of each of the first two reagents.

Similarly, the titration value of sample T_2, in which 2 ml. of each reagent except $KH(IO_3)_2$ is added in the normal Winkler order, is due to the summation of the iodine liberated or absorbed by the same factors as for T_1 except factors 2 and 6, which become: oxygen fixed from 2 ml. of each of the first two reagents, and contamination from 2 ml. of each added reagent, respectively.

Similarly also, the titration value of blank B_2, in which 2 ml. of each reagent except $KH(IO_3)_2$ is added in the reverse Winkler order, thus fixing no oxygen, is due to the summation of the iodine liberated or absorbed by the same factors as for B_1, namely, factors 3, 4, 5 and 6 except that factor 6 becomes: contamination from 2 ml. of each added reagent.

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[5] *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 819 (1934).

Thus by subtracting:

$T_2 - B_2$ = titration value of oxygen fixed from 500-ml. sample + titration value of oxygen fixed from 2 ml. of each of the first two reagents.

By doubling and subtracting further as follows:

$2(T_1 - B_1) - (T_2 - B_2)$ = titration value of oxygen fixed from 500-ml. sample.

The titration value of the oxygen initially present in the 500-ml. sample is thus obtained free from the listed recognizable errors, which cancel out in the subtraction, and can be calculated by the use of a suitable factor to milliliters per liter or to parts per million.

In this method, four 500-ml. sample bottles are filled with the water to be tested, two are fixed with, respectively, 1 and 2 ml. of Winkler reagents in the normal order, two are treated with, respectively, 1 and 2 ml. of Winkler reagents in reverse order and four titrations are made. If, in a series of tests on a particular water, the blank titration values B_1 and B_2 are found to be constant, they may be used for subsequent tests on that water and only two samples need be collected and fixed with, respectively, 1 and 2 ml. of Winkler reagents in the normal order and titrated; otherwise four samples and titrations per determination are essential.

It will be noted that the addition of the bi-iodate is for the purpose of supplying iodine in a definite and equal quantity to each of the four samples. Thus if any reducing agents are present in the sample, this iodine serves to oxidize them and yet leave an excess to be titrated. Since the same quantity of sample is titrated from each flask, the amount of iodine so used up will be the same for each titration and hence will be cancelled out in the subtraction. If the bi-iodate were not added, a particular sample might have reducing agent present in excess of the iodine liberated by the dissolved oxygen present and thus prevent the presence of the oxygen from showing up. Reducing agents are frequently met with that are oxidized much more slowly by dissolved oxygen than by iodine. An example of this is sulfide ion.

It will be noted further that the magnitude of some of the errors present in the straight Winkler procedure can be evaluated from these titrations. Thus:

$B_2 - B_1$ = Titration value of the contamination in 1 ml. portions of added reagents.

and $(T_2 - T_1) - (B_2 - B_1)$ = titration value of oxygen fixed from 1 ml. of each of the first two added reagents.

For evaluating the starch end point error, the method given by Meisenburg and Fellows,[6] applied to the proper size sample, is recommended.

Contamination in the sample can be evaluated if it is valid to assume that the interfering substances likely to be present behave in the same way to the same extent towards I_2 and KI in both alkaline and acid solution, but this point has not been thoroughly investigated.

If a contaminant is present in the sample that liberates I_2 from KI, its titration value can be determined from the titration value of blank B_1 with 1 ml. of $KH(IO_3)_2$ as given above, the titration value of blank B_2 as given above, but with the addition of 2 ml. of $KH(IO_3)_2$ instead of 1 ml. (B_3 in following equation) and the titration value of the starch end point error as determined by the procedure referred to above (S in following equation). Thus:

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[6] S. J. Meisenburg and C. H. Fellows, "The Accuracy of the Winkler Method for Dissolved Oxygen in Boiler Feedwater," Progress Report of July, 1933, for Joint Research Committee on Boiler Feedwater Studies, presented at annual meeting, Am. Soc. Mechanical Engrs., December, 1933.

$2B_1 - B_3 - S =$ titration value of the oxidizing contamination in 500-ml. sample.

Similarly, if a contaminant is present in the sample that absorbs I_2, its titration value can be determined from these same values. Thus:

$B_3 - 2B_1 - S =$ titration value of the reducing contamination in 500-ml. sample.

THEORY OF METHOD B

In the authors' closure to the discussion of their paper, Schwartz and Gurney give the following outline of the procedure for the B method:

> Two samples are collected in the usual manner. Then add the following reagents in the order and quantities listed below:
>
> | SAMPLE T_{500} | SAMPLE T_{250} |
> |---|---|
> | 2 ml. $MnCl_2$ | Same as for T_{500} |
> | 2 ml. alk. KI | Same as for T_{500} |
> | 2 ml. H_2SO_4 | Same as for T_{500} |
> | 1 ml. $KH(IO_3)_2$ | Same as for T_{500} |
>
> Add 250 ml. of untreated water to T_{250} and titrate both 500-ml. samples with 0.01 N sodium thiosulfate. Then $0.224 (T_{500} - T_{250})$ = milliliters of oxygen per liter in sample.

In this method, one 500-ml. sample and one 250-ml. sample are separately fixed with the same quantities of the same reagents added in the normal Winkler order. Before titrating the 250-ml. sample but after fixing with acid, 250 ml. of untreated sample is added.

The titration value of sample T_{500}, in which the oxygen in the 500-ml. sample is fixed, is due to the summation of the iodine liberated or absorbed by the following factors:

1. Oxygen fixed from 500-ml. sample.
2. Oxygen fixed from 2 ml. of each of the first two reagents.
3. 1 ml. $KH(IO_3)_2$ reagent.
4. Starch endpoint error.
5. Interfering substances from 500-ml. sample.
6. Contamination other than dissolved oxygen from 2 ml. of each added reagent.

The titration value of sample T_{250}, in which the oxygen in 250 ml. of the sample is fixed and to which 250 ml. of untreated sample is added, is due to the summation of the iodine liberated or absorbed by the same factors as for T_{500} except the first factor which becomes: oxygen fixed from 250-ml. sample. Hence by subtraction:

$T_{500} - T_{250} =$ Titration value of oxygen fixed from 250-ml. sample.

The titration value of the oxygen initially present in 250 ml. of the sample is thus obtained free from the listed recognizable errors which cancel out in the subtraction. In this method, three sample bottles, one 500-ml. and two 250-ml., are filled with the water to be tested, the last of which need not be collected out of contact with air. Two fixed samples are titrated, the blank correction being made automatically without the necessity for titrating extra blank samples and there is no addition of reagents in different amounts nor in reverse order.

In discussing the Schwartz and Gurney procedure, White, Leland and Button[2] object to the addition of reagents in reverse order as a blank for cancelling interferences due to the possibility of incomplete reactions. It will be noted that this objection is applicable to method A but is not valid against method B.

IMPROVEMENTS IN SAMPLING PROCEDURE

The technique employed in taking the sample for any test is of fundamental importance. It is obvious that a representative sample of the material to be tested must be taken and also that the constituent to be determined must not be introduced as a contaminant during the sampling nor during the test up to the point where such introduction will no longer cause errors. In the Schwartz and Gurney procedures, more than one sample is required and hence these separate samples must be as nearly identical as possible.

One way of meeting these conditions is to collect the samples in tandem. This technique is specified by Schwartz and Gurney. A diagram of the required sampling apparatus for method B is shown in Fig. 1. The apparatus for method *A* would be similar except that four 500-ml. Erlenmeyer flasks would be used with the last two not necessarily closed from contact with air.

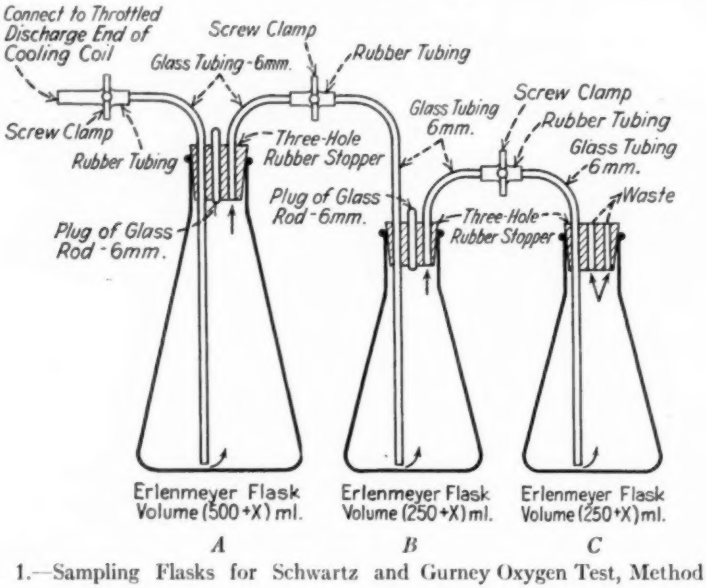
Connect to Throttled Discharge End of Cooling Coil
Screw Clamp
Glass Tubing - 6mm.
Rubber Tubing
Screw Clamp
Rubber Tubing
Three-Hole Rubber Stopper
Plug of Glass Rod - 6mm.
Glass Tubing 6mm.
Plug of Glass Rod - 6mm.
Screw Clamp
Rubber Tubing
Glass Tubing 6mm.
Waste
Three-Hole Rubber Stopper
Erlenmeyer Flask Volume (500+X) ml.
Erlenmeyer Flask Volume (250+X) ml.
Erlenmeyer Flask Volume (250+X) ml.
A
B
C

FIG. 1.—Sampling Flasks for Schwartz and Gurney Oxygen Test, Method B.

The practical use of this sampling technique results in several difficulties. Air bubbles are frequently trapped beneath the rubber stoppers and are troublesome to disengage. It is difficult to keep the rubber tubing tight enough to prevent leakage and yet not too tight to be easily removable, particularly where the pressure of water in the sampling line is not easily controllable. Similarly, the operation of the plugs is a source of difficulty, since frequently samples are spoiled, due to a sticking or to a loose plug. The contact of sample with rubber tubing and stoppers makes the sample questionable. The assembly is awkward to transfer from point of sampling to laboratory bench. The net effect is to cause the sampling procedure to be slow, with occasional accidents necessitating the taking of another set of samples.

To obviate these difficulties and annoyances, a simpler sampling procedure is proposed which meets the conditions for taking the samples outlined above by employing the principle of simultaneous sampling. A manifold is attached to the sampling line, the arms of which extend into separate bottles placed in a container so that the overflow will leave the tops of the bottles under about one inch of water. A sketch of such an apparatus for method B is shown in Fig. 2. For method A, four arms would be necessary.

The manifold shown is made of copper tubing brazed to the required shape. Other materials, such as stainless steel or even glass, could be used and also other shapes and methods of connecting. It appears best to avoid contact of the sample with rubber tubing, however, to eliminate the possibility of contamination. This may be done by connecting the manifold to the throttled discharge end of the cooling coil by means of flexible copper tubing. Another method would be to put a large loop in the copper tubing entering the manifold and insert the end into the sampling line, covering the joint with rubber tubing or tape. If a close fit is made, no appreciable contact of the sample with any material but the metal tubing will be had.

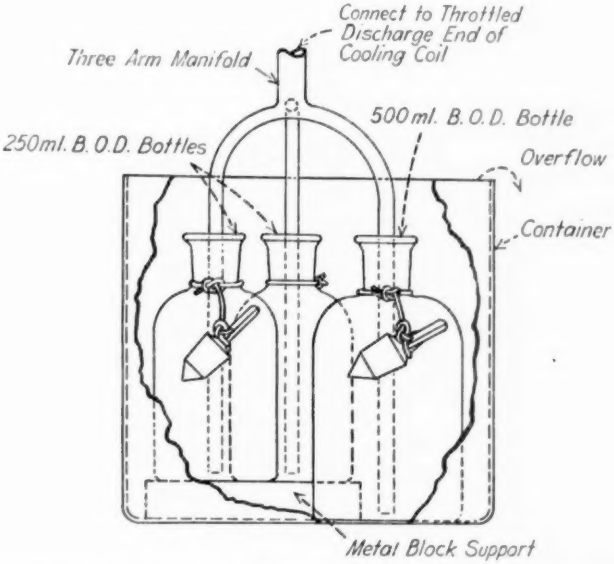

FIG. 2.—New Sampling Assembly for Schwartz and Gurney Oxygen Test, Method B.

The arrangement shown has been found convenient to use with a gallon can or a battery jar but other containers would serve just as well.

The bottles used are approximately 250- and 500-ml. capacity with narrow neck and conical-type stoppers. These are listed in chemical supply house catalogs as "B.O.D." bottles. They are ideal for the purpose since the conical tips on the stoppers fit down into the necks when the bottles are filled without entrapping air bubbles. Each bottle has its stopper wired on, since each is ground to fit. The two 250-ml. bottles in the B method rest on a support at the bottom of the container so that their necks will be level with the neck of the 500-ml. bottle.

The technique of collecting the samples in bottles immersed in the overflow water has been used by others and its benefits are particularly justified in these methods. It serves to facilitate removing and replacing stoppers during addition of reagents without exposing the samples to contact with air and, if used with the water flowing from the sampling line continuously during fixing, it also serves to wash away the manganic oxide formed outside of the bottles from the last drops leaving the pipettes.

Another improvement in the technique of sampling is to first adjust the cooling water supply of the cooling coil so as to obtain hot sample (under 212 F.) without flashing. Fill the sample bottles and allow to overflow several times with this hot sample. Then turn on the cooling water supply sufficiently to deliver cooled sample and continue flushing. The purpose is to sweep out the air in the sample bottles with hot water which will dissolve less oxygen from the air than cold water. Thus the diluting effect on subsequent fillings of the bottles, to remove the oxygen-contaminated sample resulting from the first filling in contact with air, will be more effective, since less oxygen contamination will be present. The final samples should be cooled to within five degrees of the ambient temperature of the cooling water, however, so that water will not be sucked in around the stopper due to the contraction of a hot sample upon cooling.

A source of error in the Schwartz and Gurney procedures, particularly method B, that is frequently overlooked is the adjustment of volumes in the sampling flasks. It is tacitly assumed in the A method that the respective volumes of the four sampling flasks are identical and in the B method that the volume of the larger flask is double that of the smaller flask. If these assumptions are not valid, the various error factors described above would not cancel out and the difference obtained by subtraction would not be a true value for the dissolved oxygen concentration.

Actually the real volumes of the Erlenmeyer flasks commonly used are considerably greater than the rated volumes. Calculation has shown that, if significant error is to be avoided in the methods, the real volumes of the four 500-ml. flasks should not differ by more than 1 ml. In method B, the difference in real volume between the 500-ml. and the 250-ml. flasks is not the value of 250 ml. ordinarily assumed. This introduces errors considerably greater than those which the procedure is intended to eliminate.

In either method, the possible error due to volume differences can be corrected for quite simply by adding glass beads or other inert material to the flasks in order to adjust their volumes to the desired values. In method A, it is desirable for all four volumes to be identical and then the entire contents of each flask can be used for titration, or 500-ml. portions used with the appropriate factor for calculation. In method B, the differences between the real volumes and the rated volumes of the 500-ml. flask and of the 250-ml. flask in which the sample is fixed should be made the same so that the difference in real volumes between the two flasks is exactly 250 ml., and then exactly 250 ml. of unfixed sample from the other 250-ml. flask is added for the second titration. This technique gives the titration value for a 250-ml. sample.

The glass beads or other space filler can be conveniently poured into the titrating vessel along with the sample and later returned to the proper flask. During fixing, glass beads in the flask materially aid in the mixing of added reagents when the flask is repeatedly inverted.

IMPROVEMENTS IN FIXING AND TITRATING SAMPLES

The use of potassium bi-iodate is specified by Schwartz and Gurney, but due to difficulties in obtaining this reagent readily, the author has found potassium iodate to be more satisfactory.

A source of error in the procedure is the method of adding the iodate (or bi-iodate). According to the instructions given by Schwartz and Gurney, a 0.1 N bi-iodate solution is used, and 1 or 2 ml. of it is added to each flask after fixing with acid in the same manner (by pipette) as with the other reagents. It can readily be seen that a small difference between the amounts of this comparatively strong solution added to the two samples will cause an appreciable error in the oxygen value obtained. Practically, it is extremely difficult to introduce exactly the same amount of reagent from a pipette in this manner into two separate samples.

This technique can be improved by adding the solution from a burette to the samples after they have been fixed and emptied into a casserole or porcelain dish ready to titrate with thiosulfate. Also, greater accuracy can be obtained if a larger volume of a more dilute reagent be added from the same region of a finely-calibrated burette. Consequently the author prefers to add 5 ml. of 0.01 N iodate or bi-iodate from a 10-ml. burette calibrated in 0.05 ml. to the casserole containing the fixed sample.

Another source of error that might be minimized is the titration of the liberated iodine with sodium thiosulfate to the starch-iodine end point. The use of large sample (over 500 ml.), dilute reagent (0.01 N thiosulfate) and insoluble starch (arrowroot) causes this titration to be a difficult one. If the samples are titrated at the same temperature to the same shade of end point, the error incident to the starch-iodine complex cancels out. Practically, there is considerable difficulty in matching the two end points exactly, hence an error of some definite magnitude is introduced. It can be shown readily, for example, that two porcelain dishes or casseroles have a different shade of white, and this difference is frequently great enough to make exact matching of the end points in two titrations impossible.

If starch is not used and the end points are determined electrometrically, this difficulty would be eliminated. This technique is employed by White, Leland and Button.[2a] Actually in such a titration a plot of the exact electromotive force is not needed, but rather a more simple plot of the change in electromotive force upon the addition of increments of thiosulfate. Hence a very simple potentiometer assembly can be employed.

ADAPTATION OF THE SCHWARTZ AND GURNEY METHOD B

From a consideration of the modifications in technique discussed above, the following adaptation of the Schwartz and Gurney method B for determining traces of dissolved oxygen is recommended.

Procedure for Sampling:

Using the assembly shown in Fig. 2 with volume-adjusted B.O.D. bottles, connect the manifold to the throttled discharge end of the cooling coil[7] and lower the arms of the manifold into the three sample bottles. With the water supply of the cooling coil adjusted to give hot sample without flashing, open the throttle of the sampling line enough to flow a steady but not turbulent stream of hot water into the bottles at a rate of about 1 l. per min., thus filling the 500-ml. bottle from the manifold in about $1\frac{1}{2}$ min. Allow the container to overflow about three times its volume and then turn on the cooling water fully in the jacket leaving the rate of flow of sample unchanged. Allow the container to overflow about five times its volume and slowly raise the manifold until all arms disengage from the bottles. Lower the manifold into the container with the arms outside the bottles. Stopper the bottles and examine each one for entrained air bubbles. (If any are

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[7] For preparation of cooling coil assembly, see Harold Farmer, Standard Methods of Analysis—Progress Report of Subcommittee VIII on Standardization of Water Analysis (Tentative Method for Determination of Dissolved Oxygen in Boiler Feed Water), *Transactions*, Am. Soc. Mechanical Engrs., Fuels and Steam Power Section, Vol. 51, No. 12, p. 90 (1929).

found, disengage them by swirling or with the fingers and flow more sample through all the bottles in the same manner given above about five times the volume of the container. One visible air bubble can ruin a sample.) Keep the water to be sampled flowing through the manifold and into the container. The samples are now ready to be fixed.

Procedure for Fixing:

The 500-ml. B.O.D. bottle is referred to as bottle *A*, the 250-ml. one, whose oxygen content is to be fixed, as bottle *B* and the other 250-ml. one as bottle *C*.

Add the regular Winkler reagents of the usual strength and in the regular Winkler order and quantities to bottles *A* and *B* by means of pipettes (a separate pipette for each reagent) lowered about 2 in. into the bottles (except the acid, which should be released in the necks of the bottles) while the bottles are under the surface of the liquid in the container. Add no reagents to bottle *C*. Before lowering the pipettes into the bottles, allow a drop of liquid to flow out and hang on the tips of the pipettes each time, so that an air bubble will not be introduced.

After the addition of the manganous ion and alkaline iodide solutions, stopper bottles *A* and *B*, remove from the container, and mix the contents thoroughly by repeated inverting. Then replace them in the container, allow to stand until the precipitate has settled (about 10 min.), unstopper, fix with the acid, stopper and again mix by inverting. Remove all of the bottles from the container preparatory to titrating.

Procedure for Titrating:

Hold all samples at 70 F. or lower. Pour the entire contents of bottle *A* (including glass beads) into a large porcelain dish or casserole. Rinse out once with a few milliliters of distilled water. Add exactly 5 ml. of 0.01 *N* potassium iodate (or bi-iodate) from a 10-ml. burette. Titrate with 0.01 *N* sodium thiosulfate, from a 5- or 10-ml. burette preferably calibrated in 0.01 or 0.02 ml., to the starch end point, adding 2 ml. of arrowroot starch solution when close to the end point. Stir vigorously during titration, particularly when near the end point. Record titration value in milliliters as T_A.

Clean out this porcelain dish or casserole (returning glass beads to bottle *A*) and pour the entire contents of bottle B (including glass beads) into it. Rinse out once with a few milliliters of distilled water. Add exactly 250 ml. of unfixed sample from bottle *C* to the dish or casserole. Add exactly 5 ml. of the same 0.01 *N* potassium iodate (or bi-iodate) from the same region of the same burette. Titrate to the starch end point in the same manner as given above. Record titration value in milliliters as T_B.

Clean out the porcelain dish or casserole (returning glass beads to bottle *B*). Clean out all bottles and set up preparatory for next test. If desired, the sampling water may be allowed to flow continuously so that a sample will be ready when it is needed.

Calculation of Results:

By adjusting the volumes of bottles *A* and *B* as previously described, the difference between the volumes of these two bottles when stoppered will be exactly 250 ml. Hence, the difference between the titration values T_A and T_B will be the titration value of a 250-ml. sample. Hence:

$(T_A - T_B) \times (0.32) =$ parts per million of O_2 in sample,

or,

$(T_A - T_B) \times (0.224) =$ milliliters of O_2 per liter in sample.

RECOMMENDATIONS FOR FURTHER WORK

It is desirable, in determining the accuracy and sensitivity of a method, to compare it with other methods that are believed to be comparable in these regards, by using all methods on a series of identical samples. This is particularly necessary with a method for determining traces of dissolved oxygen due to the difficulty in obtaining a standard sample. Hence, a number of such comparisons on identical samples would be necessary to set the limits of accuracy and of sensitivity of the three methods discussed here.

Since contaminants in the sample are always likely to be present in varying amounts, some data are necessary to determine what limits can be tolerated by each method. This is particularly true for the common contaminants such as sulfite and nitrite. Such information would answer several perplexing questions that have been raised particularly in regard to the two above-mentioned materials.

The benefits of electrometric titration of the fixed samples over the usual titration to the starch end point have been pointed out by White, Leland and Button.[2] However, the equipment required is too expensive and not simple enough to meet with very wide plant use. It is believed that considerable simplification can be made in this equipment which will reduce its cost and increase its use in all procedures utilizing the thiosulfate-iodine titration.

Acknowledgment:

The author wishes to thank the many investigators who have contributed to and encouraged this work by their valuable suggestions and criticisms. Most of the material here presented was adapted from observation in the field of difficulties encountered in utilizing the various methods of analysis.

DISCUSSION

Messrs. M. C. Schwartz[1] and W. B. Gurney[1] (*presented in written form*).—The following remarks prior to a discussion of Mr. Daugherty's paper are *apropos*. When the manuscript was being prepared for our first paper[2] on the determination of dissolved oxygen, it was decided, in deference to the Joint Research Committee on Boiler Feedwater Studies under whose auspices research work was being carried out at the University of Michigan, not to formulate definite apparatus, sampling devices and the like for the determination of dissolved oxygen. The first paper therefore was essentially theoretical. Shortly afterwards a need for a procedure became evident so one was incorporated in our second paper.[3]

Mr. Daugherty's note under *Theory of Method A* is fully covered in the body of both of our papers. Under *Improvements in Sampling Procedure*, Mr. Daugherty states "the practical use of the sampling technique results in several difficulties." We are of course not prepared to state that our procedure is the best possible one; however, obviously we have not experienced the difficulties described. The "source of error" in the difference between the rated and actual volumes of the Erlenmeyer flasks can be simply considered by the use of a factor, or still better, the use of McLean sampling tubes as described in our paper.[3] Mr. Daugherty states that "a source of error" in our procedures is the use of a 0.1 *N* bi-iodate. Just how he arrived at this conclusion we are at a loss to understand since it is plainly stated in both our papers that we used 0.01 *N* bi-iodate in all titrations.

The determination of dissolved oxygen shows little sign of becoming standardized. It is not the intention of the writers to advocate their methods or procedures to the exclusion of others. In this instance, in justice to themselves, they must point out that the "several sources of error" described by Mr. Daugherty are actually non-existent as a reading of both papers would disclose.

[Editor's Note.—In addition to this discussion of the Daugherty paper, Messrs. Schwartz and Gurney transmitted the following note intended for the information of the industry:

It has been called to our attention that an error is being made in the determination of dissolved oxygen by the writers' method.[2] Potassium bi-iodate should be added in the titrating vessel after the sample is "fixed," not in the sampling flask.

Although the procedure and, in particular, the above point, have been described in greater detail in *Bulletin No. 21* of the Louisiana State University Press,[3] the apparent limited circulation of this publication has made it advisable to call it to the general attention of those using or contemplating using our method for determining dissolved oxygen.]

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[1] Research Chemist, and Efficiency Engineer, respectively, Louisiana Steam Generating Corp., Baton Rouge, La.

[2] M. C. Schwartz and W. B. Gurney, "The Determination of Traces of Dissolved Oxygen by the Winkler Method," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 796 (1934).

[3] M. C. Schwartz, "Dissolved Oxygen in Boiler Feedwater," *University Studies No. 21*, Louisiana State University Press, Baton Rouge, La. (1935).

MESSRS. C. H. FELLOWS[4] and J. M. REYNAR[4] (*presented in written form*).—The writers appreciate this opportunity to recognize Messrs. Schwartz and Gurney for the ingenuity that led them to attack the problem of the determination of dissolved oxygen from a new angle. Although the procedure as originally published was far from perfection it stimulated widespread study of the double titration idea as applied to this determination until at the present time their B method, modified in a minor although very important way, can be used with assurance of precision impossible with the Winkler method as it has been commonly used for the determination of very small quantities of oxygen in boiler feedwater.

To the author of the paper under discussion we believe should go the principal credit for making the B method of Schwartz and Gurney a workable procedure. Mr. Daugherty immediately recognized the merit of the method, and by diligent study so analyzed it that he has been able rationally to point the way to successful manipulation. To those who have doubted the workability of this B method may we recommend the detailed analysis of the theory of that method as presented by Mr. Daugherty.

Aside from this B method of Schwartz and Gurney, the only precision method that has been proposed in recent years for the determination of dissolved oxygen in low concentration is that offered by White, Leland, and Button, and this method is perhaps the one of greatest fundamental accuracy. Irrespective of the nature of the water and nearly all ions interfering with the Winkler reaction, this method can be used with the assurance of correct results. It has, however, as its greatest objection a complexity of apparatus and manipulation that necessitates using it as a strictly laboratory procedure. It is not portable and cannot readily be moved from plant to plant.

The B method of Schwartz and Gurney is theoretically sound and is very attractive from a manipulative standpoint. Actual application of the procedure recommended has revealed the necessity for a modification of the sampling vessels, to get away from the difficulties attendant upon the use of rubber stoppers in the flasks. While samples were being taken, bubbles persisted in lurking under the stoppers. Upon sealing the flasks, slight elevations in temperature usually caused the stoppers to pop out. Slight decreases in temperature created a vacuum which rendered the plug unremovable from the hole through which reagents should be added. Indeed, a successful removal of a plug usually was accompanied by the introduction of an air bubble which

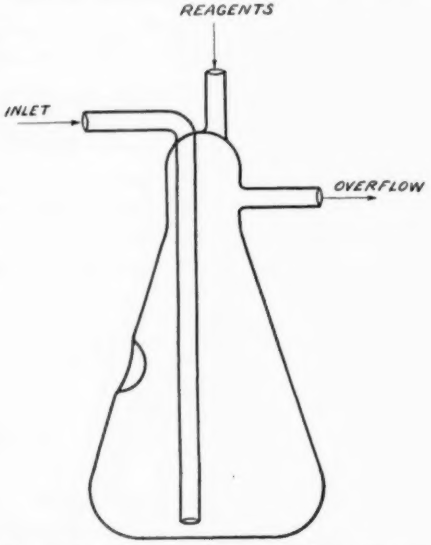

FIG. 1.—Sampling Flask for Dissolved Oxygen Determination.

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[4] Head of Chemical Division, and Engineer, respectively, Research Dept., The Detroit Edison Co., Detroit, Mich.

ruined the determination. The presence of glass beads for space fillers to adjust the volume to 500 and 250 ml. was likewise found to be a nuisance.

The modification in sampling procedure recommended by Mr. Daugherty appears to be a decided improvement over the stoppered flasks, but the overflow of the outer container would be difficult to cope with in most power plants. The subsequent opening of the bottles for the addition of reagents has proved satisfactory for plant tests, but could not be recommended for precision measurements.

Sampling flasks which have proved very satisfactory in the writers' laboratory were prepared from 500 and 250-ml. Erlenmeyer flasks (Fig. 1). They are essentially the same as those of Schwartz and Gurney with the exceptions noted. The neck of the flask is sealed around the inlet tube and two other outlet tubes are sealed on. One near the top serves as an overflow, whereas the other is located at the apex of the dome-shaped top and is used for the introduction of reagents. A short rubber tube is fitted to this latter tube and sealing is effected by means of a screw clamp. During the filling operation all air escapes through this open vent which is pinched off after water has begun flowing. Reagents are added through this opened tube by introducing fine-tipped calibrated pipettes into the body of liquid. The small amount of solution in the flask exposed to air is pinched off and discarded during the fixing procedure.

The volumes of the sampling flasks are adjusted to exactly 500 and 250 ml. by blowing a concave or convex bubble on the side of the flask softened in an oxygen flame.

As for the effect of rubber tubing on the sample line and flasks, the writers have been able to detect no influence of rubber upon the results for dissolved oxygen even at zero oxygen concentration.

The Winkler reagent, $Mn(OH)_2$, was found to be effective beyond the accuracy of other factors of the test in the removal of dissolved oxygen.

Experiments carried out in this laboratory indicated that the use of starch as indicator introduced a variable error in the reduction titration in the neighborhood of 0.01 ml. per liter of apparent oxygen. The end point appeared too soon. It is not believed possible to achieve the greatest accuracy even with the error-compensating procedure of Schwartz and Gurney so long as the erratic starch indicator is used in the reduction titration.

Mr. Daughterty for the first time presents the reason for the addition of a definite quantity of oxidizing solution before the final titration of the fixed oxygen solution. Heretofore it seemed analogous to the case of a man weighing a pin on a rough balance by sticking it into a pin cushion, weighing pin and pin cushion and subtracting the weight of the cushion. To those of us who determine oxygen on only pure condensate it still seems analogous, for, after all, the impurity being investigated is dissolved oxygen, not a reducing substance. This procedure merely establishes that reducing substances are present, thus invalidiating the determination without correcting for them.

The writers have found electrometric titrations to be practicable and extremely accurate. Changes in solution potential are followed by means of a potentiometric circuit involving a calomel half-cell and a platinum electrode dipping into the fixed solution in an open vessel. Thus the errors of the starch end point and the necessity for making the addition of a definite quantity of oxidizing solution are eliminated. The presence of reducing substances is immediately indicated by an initial reading of the solution potential, and the titration end point is indicated by the greatest change in potential per unit addition of titer.

Electrometric titrations are, of course, not practicable at all plants or points where dissolved oxygen is measured. Such a procedure, however, can be carried out in any well equipped laboratory and offers a precision check on the routine determinations using starch as an indicator.

Potassium iodate, KIO_3, is preferred by the writers to the bi-iodate, $KH(IO_3)_2$, although both are very satisfactory as oxidants.

The writers have prepared a comprehensive résumé of the investigations carried out in their laboratory concerning methods for detecting dissolved oxygen. This will be released for publication in the near future.

MR. R. C. ADAMS[5] (*presented in written form*).[6]—The primary interest of any engineer in the report of another's work lies in the application of that work to his own needs. Therefore I shall discuss Mr. Daugherty's suggestions as they might be applied to the determination of dissolved oxygen in the feed systems of Naval vessels. First, I should like to comment on his criticisms of the procedures of Schwartz and Gurney, then to discuss his proposed procedure which is based on the Schwartz and Gurney method B, and finally to make a brief presentation of our procedure at the Naval Engineering Experiment Station, which also has been developed from method B.

In the first place it seems that Mr. Daugherty's opposition to the use of rubber stoppers and rubber tubing is rather excessive. There is difficulty in removing bubbles of air trapped beneath stoppers, particularly the stoppers of wide-mouth flasks such as we use. We have found that a short length of copper wire is useful in dislodging these trapped bubbles when a stopper is first used. Once the under surface of the stopper has been wet, we have found no further trouble. If the stopper is put into the flask at an angle until wet, it then may be seated firmly without trapping air. I agree with Mr. Daugherty as to the undesirability of a plugged hole in the stopper as a means of adding the Winkler reagents. It is almost impossible to add constant volumes of the reagents and to avoid either leaving a bubble of air within or below the hole, or wasting part of the reagent on top of the stopper. We have had no difficulty with leakage at joints between rubber and glass tubing. If the outlet end of the rubber tubing is at a higher level than the top of the flasks, so that there can be no siphoning action, there exists no differential head to force in air. Finally, it seems improbable that rubber stoppers and tubing which have been boiled for hours in strong caustic solution could release sufficient sulfur into the sample to affect the results. This seems the more improbable when it is considered that the same rubber fittings are used repeatedly, that sampling requires an extended flushing period, and that the samples remain in the flasks in contact with the rubber for a very brief period before the iodine equivalent of the dissolved oxygen is fixed by acid. We should be very glad to hear of definite data on this point since it would be extremely difficult for us to avoid the use of rubber in our sampling.

The author comments that the assembly is awkward to transport. While this is true, it is not forbiddingly so, and

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[5] Chemical Engineer, U. S. Naval Engineering Experiment Station, Annapolis, Md.

[6] The opinions herein expressed are those of the discusser and are not to be construed as official opinions of the Bureau of Engineering or of the Navy Department.

probably is no more difficult than the alternative which he proposes. We recently have completed a dissolved-oxygen survey on a destroyer during which the 65 samples were collected in the engine room, placed in a padded pail, and carried safely up ladders and down stairs to the temporary laboratory. No samples were lost *en route*, but one was lost when a careless engineman kicked over a sampling train which had been set up on the floor grating.

In connection with the author's suggested procedure, I offer my unqualified approval of simultaneous sampling. We have used tandem sampling, that is, a sampling train, and have observed its deficiencies. There is a variation in the dissolved-oxygen content of the boiler feedwater on Naval vessels, which results from the changing conditions brought about by the maneuvering of the ship. If the oxygen concentration is decreasing, the first flask of the train, which is the 250-ml. flask, will have a lower concentration than the second. The result calculated from the difference between the two titrations will be greater than that indicated by either titration, and will be erroneously high. The reverse, with erroneously low results, will occur when the oxygen concentration is increasing. In such cases we have been forced to sacrifice the advantages of the duplex procedure and calculate a "most probable" result from the average of the two individual titrations. It should be noted that simultaneous sampling will not eliminate this source of error unless the rate of flow to the 250-ml. sample is exactly half the flow to the 500-ml. sample. But even without this added precaution the possible error would be minimized.

The use of immersed sampling bottles should be very effective in preventing atmospheric contamination of the samples. Unfortunately such a system scarcely could be used on shipboard. Unless the sample container were made with inconveniently high sides, the ship's motion would induce swashing and slopping of the contents, which cannot be tolerated. Sampling waste on shipboard must be confined and conducted directly to the bilges instead of being permitted to find its own way downward over magnesia insulation and electrical conductors. Also it would not be feasible to carry the Winkler reagents to the dispersed sampling locations, as would be possible in a shore station.

It is suggested that the adjustment of volume of the titrated samples might be simpler if made by measuring the desired amount from the sampling bottles rather than by an exact adjustment of their individual volumes. This would be particularly so if a number of sample bottles were used. The time for pipetting a sample from a bottle would be little if any greater than would be required for washing out the bottle. The volumetric error introduced by failure to titrate the entire sample would be much less than the inappreciable one from displacement of sample by addition of the Winkler reagents. Probably it would be less than 1 per cent.

The use of iodate or bi-iodate probably is unnecessary unless the dissolved-oxygen concentration is so low that an occasional zero result is obtained, or reducing constituents are known to be present in the water. We have had no experience with the latter condition, but we would appreciate information concerning the usefulness of iodate in determinations of dissolved oxygen in sulfite-containing waters.

The method of dissolved-oxygen determination which we are using in our work on Naval vessels remains essentially as it was described at last year's annual meeting of this Society except that we now use electrometric titration instead of colorimetric titration. The following procedure is used. The cooled sample is collected in a train of three flasks, the first of which is of 250-ml. and the other two of 500-ml. capacity. Three stoppered-tip pipettes, containing the Winkler reagents, are fitted through the stoppers of each of the first two flasks. The flow of water is continued until the entire train has been flushed with 15 to 20 complete changes of water. The train is then removed and Winkler reagents are added to the first two flasks on the spot, without removing the stoppers or otherwise exposing the sample to the atmosphere. The fixed samples are carried to the laboratory. Five hundred milliliters from the second flask are titrated, and then a second 500-ml. sample made up of 250 ml. each from the first and third flasks. Although electrometric titration is used, starch is added to the titrated samples as a check. The result is calculated from the difference between the two titrations in accordance with the Schwartz-Gurney procedure B.

We employ a Beckman pH meter, together with calomel and platinum electrodes for the measuring instrument of the electrometric titration. The arbitrary pH scale of the instrument is used with a standard setting of the temperature regulator, but the readings are not calculated to electromotive force although such a conversion could be made. Preliminary testing of this arrangement showed that an excellent curve of milliliters of thiosulfate *versus* scale reading was obtained. This relationship is essentially linear between readings of 2.25 and 3.25, and in titrations of samples having a high oxygen concentration the linear relationship extends above and below these limits. In a plot of duplex titrations the linear portions of the two curves are parallel so that the difference is constant. Therefore the result calculated from this difference is constant. Accordingly we have adopted a scale reading of 2.75, the mid-point of the linear portion, as the standard end point of our electrometric titrations. In each titration it is necessary to obtain only two readings: one between 2.25 and 2.75, and another between 2.75 and 3.25. A linear interpolation between the milliliters required at the two points on the titration curve is made to determine the amount equivalent to a scale reading of 2.75. This interpolation usually can be made mentally. The dissolved-oxygen concentration of the sample is calculated from the difference between the interpolated values for the two titrations. An analyst can make a determination unassisted by the use of a measuring pipette instead of the burette for the thiosulfate and a motor stirrer in the sample being titrated.

This procedure has been found quite satisfactory on shipboard. The standard Beckman 5-in. electrodes were insufficiently sturdy for shipboard use, but this fault is being corrected in special electrodes which the manufacturer now is making for the Station.

We shall continue to use and to develop the basic procedure outlined above. We may find it possible to adapt Mr. Daugherty's suggestion of a simultaneous sampling to our somewhat unusual conditions. I hope that it will be possible to make a complete report on our method and the results which are obtained within the next few months.

MR. J. D. YODER[7] (*by letter*).—Daugherty concedes the claim of White, Leland and Button that Schwartz and Gurney's method A may not correctly

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[7] The Permutit Co., New York City.

measure all interferences because the reagents are added in their reverse order, but he contends that the B method, in which the reagents are added in their normal order, should be accurate in cancelling out the interferences. This later conclusion seems questionable because there is no assurance that the treated 500-ml. A sample and the treated 250-ml. B sample, which is subsequently diluted with the untreated C sample should be affected identically by interfering substances.

Undoubtedly the determination of the end point electrometrically as proposed by White, Leland and Button is more sensitive than the starch indicator, but as the starch end-point error is canceled out by the double titration, the greater complication of the potentiometer does not seem warranted. During the past few months we have used the potentiometer method in the field for determining small traces of oxygen in water from a deaerating hot lime soda softener but found no advantage from its use to justify its greater complication. The Schwartz and Gurney method B, with such modifications as proposed by Daugherty, seems to be the most acceptable method that has yet been offered.

Mr. T. H. Daugherty[8] (*author's closure, by letter*).—The discussion by Messrs. Schwartz and Gurney may best be answered by citing a bit of history. The first published account of Messrs. Schwartz and Gurney's work on dissolved oxygen determination[2] was widely read and discussed in the field. It was found to be a new approach which gave promise of greater accuracy and came at a time when accuracy in this test was in demand. A number of attempts were made to apply the method in the field to the problems for which such accuracy was needed, but with indifferent success. It was soon found that Messrs. Schwartz and Gurney had limited themselves to what might be termed an architect's sketch, whereas detailed working drawings were necessary for successful application in the field. Within the author's knowledge, five individuals, recognized in the field, independently arrived at four different interpretations of Schwartz and Gurney's method A of cancellation of Winkler errors by double titration. Comparison of course revealed weak points in all four.

The author and others well appreciated that here was a diamond in the rough, but for its full value to be realized it would require interpretation and reduction to practice so that the accuracy and advantages claimed for it could be attained by the competent operator, in whose hands and upon whose samples the practical value of any analytical procedure must depend.

The first attempt to meet this field need of a practical procedure was the author's discussion on the procedure for method B[9] which was followed by Messrs. Schwartz and Gurney's closure to the discussion of their paper in which their procedures for methods A and B were similarly outlined. This was followed by Mr. Schwartz's second paper on the subject[3] which was not available to the author prior to the presentation of his paper, presumably due to a very limited circulation. A subsequent examination of it reveals a further clarification of the procedure, the presentation of sampling apparatus and minor changes, such as the external addition of 0.01 *N* potassium bi-iodate instead of the original internal addition and specifi-

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[8] Research Chemist, Hall Laboratories, Inc., Pittsburgh, Pa.

[9] Discussion by T. H. Daugherty of paper by M. C. Schwartz and W. B. Gurney, "The Determination of Traces of Dissolved Oxygen by the Winkler Method," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 819 (1934).

cation of 0.1 N reagent (solution No. 6 in the first paper[2]).

Meanwhile the attempts to apply the method in the field had continued and it was observed that difficulties were still encountered and that there was still a general lack of understanding of the theory of the method. As reports and personal observations of data and difficulties accumulated, and with an earnest desire to make the application of the Schwartz and Gurney principle as successful in use as it was sound in theory, the author presented this paper. If it produces a clearer understanding of the Schwartz and Gurney principle and gives a simple, workable procedure, the author believes it to be worth while.

Messrs. Fellows and Reynar have made a very thorough study under field and laboratory conditions, and present as a modification to the author's adaptation of the B method an ingenious sampling arrangement. This should be practical, although the author preferred to steer clear of special glass apparatus because of difficulty of replacement when it is broken. A one-gallon metal can, copper manifold and standard B.O.D. bottles, volume-adjusted with glass beads, was the simplest arrangement that the author could devise that would normally be satisfactory. The principle of hot-flushing and addition of reagents under water still seem sufficiently desirable to overcome the objection to overflow in a plant, and seem just as accurate as the proposed change.

Mr. Adams deserves considerable credit for devising apparatus and adapting the procedure to use on shipboard. The unusual difficulties mentioned were not considered by the author, since they are not normally met with.

In addition to the discussion presented here, Mr. Lima has called attention to two errors in the text of the paper which have been corrected in this printing. Likewise valuable criticism has been received in letters from Messrs. Hecht, McDermet, Joos and Theriault that does not appear in the discussion.

In conclusion the author believes that a procedure for determining dissolved oxygen has been devised and presented which (1) employs the Winkler principle of fixing and titrating and the Schwartz and Gurney principle of double titration to cancel errors otherwise introduced; (2) is practical for use by field operators on field samples; (3) is essentially free from the errors incident to the regular Winkler method; (4) contains a new principle of sampling; and (5) is more sensitive and capable of greater accuracy than other field methods in use.

DYNAMIC TESTS BY MEANS OF INDUCED VIBRATIONS

By Rudolf K. Bernhard[1]

Synopsis

This paper gives a short report of a method of dynamic research in the engineering field. Various applications of this method are described. Tests have been carried out since 1928, the system being used for various types of investigation, from small laboratory specimens to heavy ships. Different materials and all types of construction have been examined and compared by fatigue and aging tests. A few of the results, particularly various damping and amplifying factors of standard engineering systems are considered. Advantages and difficulties of this method are summarized.

In the method here discussed, vibration is induced by centrifugal forces resulting from revolving eccentric disks. As soon as the natural period of the structure coincides with the revolution of the disks, the amplitude of vibration of the system and the power input to drive the revolving disks reaches a maximum. Plotting the power input against the revolutions of the disks, characteristic curves can be obtained, which indicate first of all the natural frequency of the vibrating system which is to be investigated. Any coincidence between natural frequency and dynamic load, such as a locomotive with unbalanced masses passing over a bridge with critical speed, is to be avoided. Vibrating a structure with this type of oscillator for a long period has in many cases a similar effect as an annealing process; internal stresses remaining from the rolling process or due to welding are in some measure dissipated over the complete structure. A change in the natural frequency may indicate some failure or plastic flow; this forms a means to check from time to time the behavior of the structure, even after erection.

The determination of the natural frequency of soil and the propagation speed of these induced vibrations can sometimes replace seismic investigations or borings.

Introduction

Striking a piece of earthen ware and listening for the resulting tone to die out is the most ancient dynamic impact investigation by means of free vibration. This method has been used for centuries to discover whether any cracks exist. In the practical field of the engineer, static tensile strength and elongation tests have been supplemented in recent years by corresponding dynamic tests (**1**)[2] using fatigue, endurance limit, and damping ductility (crackless plasticity) (**2**) testing machines. These machines (**3**) are adapted only to the testing of standard specimens, and so are limited

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[1] Consulting Engineer, Baldwin-Southwark Corp., Philadelphia, Pa.

[2] The boldface numbers in parentheses refer to the reports and papers given in the list of references appended to this paper, see p. 644.

in scope. Tests on actual structure, however, will result in a more complete analysis of existing conditions. Furthermore, failures usually occur in the connections of different members, often due to secondary stress effects, and rarely do failures occur because of defects in the member itself. The usual tests will give no indications in regard to such failures. Fatigue and damping tests of large members, including their connections, are expensive, if not altogether impossible. The dynamic method, described in this paper, using artificial vibrations produced by means of special oscillators, permits of tests to be made on structures of different size from a small specimen to a large actual structure. This method has been used for several years with increasing success in various countries, by the U. S. Coast and Geodetic Survey (4), and the U. S. Navy, in a number of different laboratories for testing materials (5) (Berlin, Dresden, Zuerich), by railroad companies in inspecting bridges (6), rolling stock, maintenance of way, etc. (Germany, Switzerland, Italy and Russia), in the aviation field (7) (Deutsche Versuchsanstalt für Luftschiffahrt, Berlin), and for soil investigations (8) (Degebo-Seismos-Berlin). Since only a small amount of energy is used, the method is economical; instead of forced vibrations, free vibrations, more or less near the resonance point, may be applied. Certain dynamic properties, not only of materials as such but of complete structures, new or old, finished or unfinished, including outside connections, can be determined in many cases. The fundamental principle is simple. The testing machine, the oscillator itself, is relatively foolproof, so that an unskilled workman can handle it. External conditions, such as rain and moisture, have practically no influence and tests can be made in the field almost as successfully as in the laboratory. Tests can be readily repeated which can seldom be done with moving live loads.

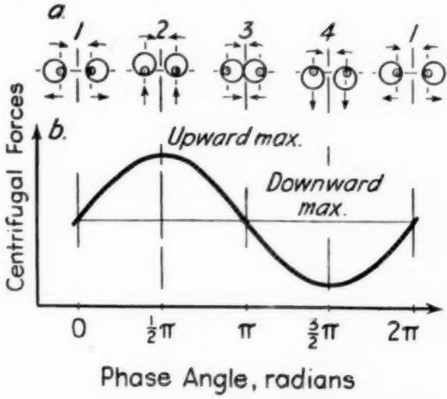

FIG. 1.—Exciting Artificial Vibration.

(*a*) Position of the two rotating masses during one revolution.

(*b*) Corresponding centrifugal forces alternating in a sine form.

Cases 1 and 3: horizontal forces neutralized.

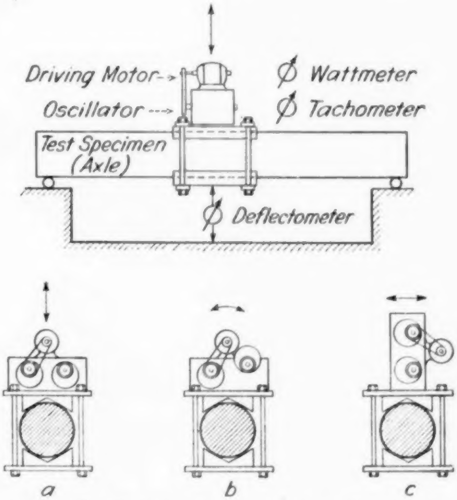

FIG. 2.—Schematic Diagram of Standard Oscillator Test.

(*a*) Bending test.

(*b*) and (*c*) Torsion test.

In (*b*) the two masses have a difference in phase of 180 deg.

OSCILLATORS

Oscillators have been built for frequencies between 1 and 100 cycles per second and with centrifugal forces between 1 and 50,000 lb. Assuming an amplifying factor of 40 (bridge structure), the largest alternating force of a heavy oscillator may become 40 × 50,000 or 2,000,000 lb. Each oscillator consists of two revolving disks (Fig. 1), an electric motor to drive these disks and two measuring instruments, one to measure the frequency of the disks and the other to measure the power input (watts) of the motor. For more accurate results a special motor-generator set (Ward-Leonhardt System), giving a closer regulation of the frequency, is desirable. To change the frequency, the resistance in the field circuits of the motor-generator set are varied, thus giving a gradual and exact regulation of the voltage of the oscillator motor. To change the centrifugal forces, the eccentricity of the revolving masses is suitably adjusted. In speeding up the motor, the centrifugal force, Z, increases directly as the square of the cycles:

$$Z = Mr\,\omega^2$$

where M = mass,
r = eccentricity,
ω = angular velocity.

For torsion tests, the eccentricity of the two masses can be changed so that they have a difference in phase of 180 deg. (See Fig. 2(*b*).)

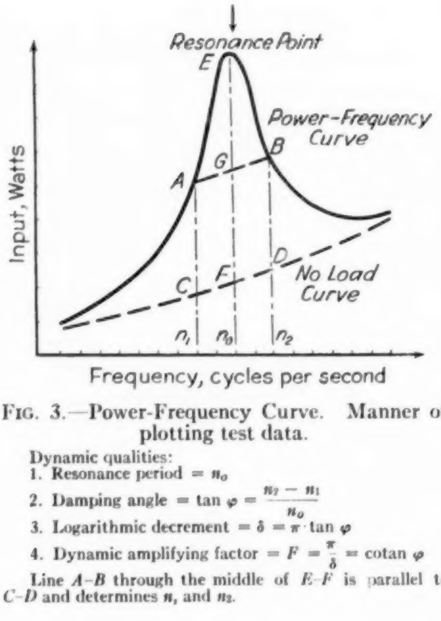

FIG. 3.—Power-Frequency Curve. Manner of plotting test data.

Dynamic qualities:
1. Resonance period $= n_o$
2. Damping angle $= \tan\varphi = \dfrac{n_2 - n_1}{n_o}$
3. Logarithmic decrement $= \delta = \pi \cdot \tan\varphi$
4. Dynamic amplifying factor $= F = \dfrac{\pi}{\delta} = \cot\varphi$

Line *A–B* through the middle of *E–F* is parallel to *C–D* and determines n_1 and n_2.

TEST PROCEDURE[3]

The standard test procedure is as follows: The oscillator is rigidly fixed to the vibrating system (Fig. 2). The frequency is slowly raised, starting with a low value. For each frequency the corresponding power (electrical input) has to be plotted in order to get the frequency-power curve (Fig. 3). Instead of the input, the corresponding deflections, stresses, speeds, accelerations or phases may be measured. After having gone through the resonance points (one or even more) for a certain number of frequencies, the same curve can be plotted in the reverse direction by slowing down the speed of the oscillator, thus checking the first curve. If the

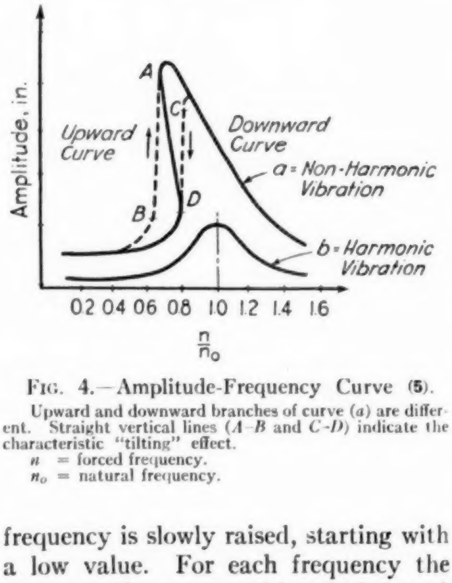

FIG. 4.—Amplitude-Frequency Curve (5).

Upward and downward branches of curve (*a*) are different. Straight vertical lines (*A–B* and *C–D*) indicate the characteristic "tilting" effect.
n = forced frequency.
n_o = natural frequency.

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[3] For theoretical principles of testing see Appendix, p. 643.

two curves do not coincide, it may be due to non-harmonic vibration. Nonharmonic vibrations are caused, when, with varying amplitudes, the dynamic qualities, that is, the elastic capacity, the damping values, or the oscillating masses are changing likewise. Figure 4 shows the upward and downward amplitude-frequency curve of such nonharmonic movements, demonstrating by the two vertical straight lines (*A–B* upward curve and *C–D* downward curve) within each branch the characteristic "tilting" effect of unstable equilibrium. From the author's point of view, this may be considered as a rather convenient way to prove the existence of nonharmonic mechanical vibrations, which cannot be so easily detected by other methods. Further details regarding this phenomenon are described elsewhere (5).

PRACTICAL APPLICATION AND RESULTS

General Tests:

The nearer the resonance point of the oscillating system at which the tests are carried out, the smaller the required energy input and the more economical the investigation will be. A large engineering structure can be tested with this resonance method, even after erection in the field. All tests with completed structures have the advantage that not only the internal damping influence of the material, but also the other conditions (connection, foundations, soil, etc.) can be studied—conditions which are always important in practice. The value of all these investigations may be considered, therefore, in the actual imitation of service conditions.

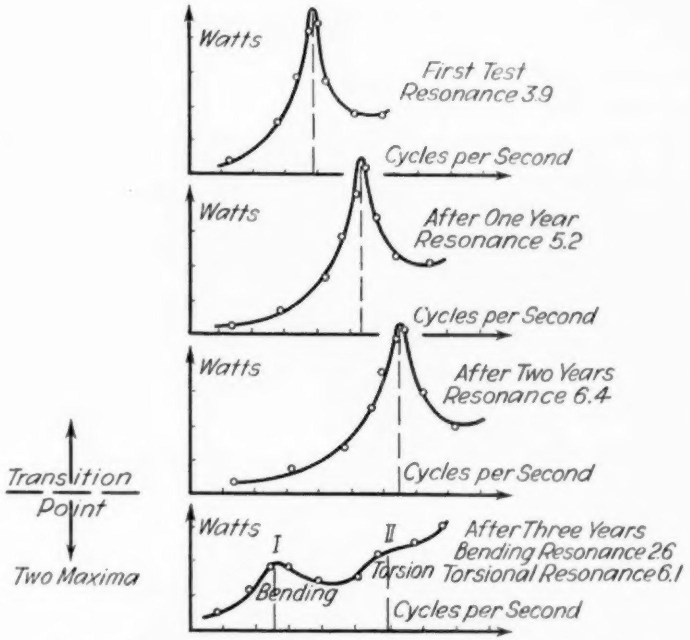


FIG. 5.—Dynamic Survey by Means of Power-Frequency Curves.

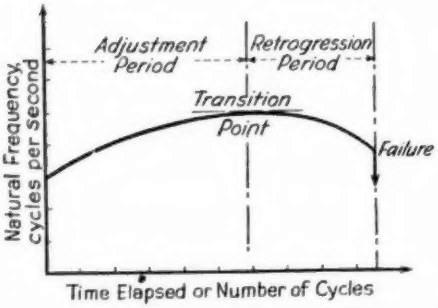
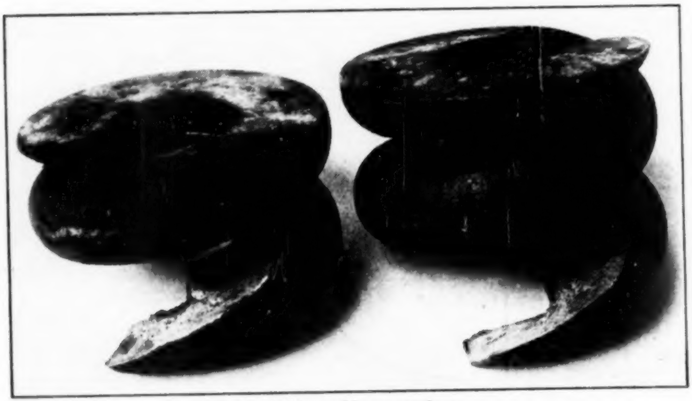
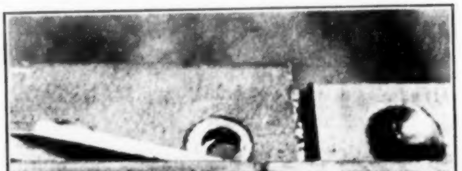


FIG. 6.—Time or Aging Effect. Natural frequency-time curve.



(a) Failure of heavy springs.



(b) Failure of riveted connection.

FIG. 7.—Result of Oscillator Fatigue Tests.

Fatigue Tests:

The fatigue effect (retrogression) of an oscillating system (old bridge) can be determined by plotting the power-frequency curves periodically (annually) Fig. 5. The natural frequency rises generally in the first few years after erection, for instance (the so-called period of adjustment) and falls in the later years preceding failure (Fig. 6). The following tentative explanation might be given for this phenomenon. During the adjustment (or "training") period, the system becomes more and more rigid, the connections settle and stiffen, and the safety of the structure increases. Later this effect ceases, the connections soften, and, as soon as any weak spot develops—this may be only a minute crack, invisible under the microscope or a localized plastic flow—the resonance point drops (5). This dropping may be at first only 1/100 of a cycle per sec. It has been possible in several cases to determine by this method the approach of the danger point of certain oscillating systems, which had been worn out and had to be renewed. This is of importance for example in the case of old bridges, where a decision must be made as to whether to strengthen or to renew the structure entirely, assuming that neither the supports nor the foundations are causing this change in the natural frequency. In several cases, where a change of the resonance point has been caused by alterations in the foundations, this has been detected by determining changes in the vibrating mass of the complete structure—its foundations included (5).

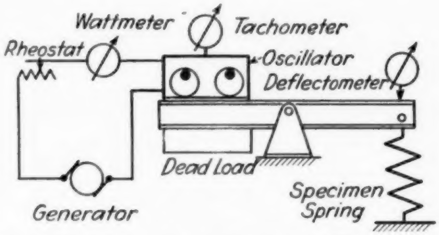


FIG. 8.—Oscillator Arrangement of Equipment for Direct Stress Test.

Annealing Steel by Means of Vibration:

Vibration of a completed structure produces a certain stress relief, similar to annealing by causing localized plastic distortions to take place (5). Internal or concentrated stresses such as those remaining in the welded connections of a structure due to heat or in structural shapes of steel or aluminum due to rolling, have thus been relieved by vibration. Vibrating a structure for a fixed time may be substituted, therefore, in some measure for an expensive annealing process. Quite a number of welded railroad bridges in Germany have thus been stress relieved.

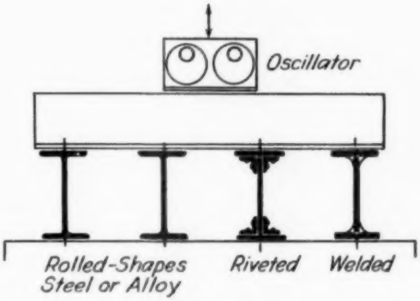


FIG. 9.—Simultaneous Comparison Tests on Various Structures.

Laboratory Tests:

In laboratory fatigue tests on small specimens (such as bars, rods, axles, crackshafts, springs, Fig. 7 (*a*), or riveted, Fig. 7 (*b*), and welded joints), a cantilever arrangement (Fig. 8) or a simple truss or girder (Fig. 2) has been used. The desired initial load is applied by means of a special loading platform, simulating the dead load. These systems have been in use for several years in material testing laboratories in

Europe (5). With standard stress (or deflection) measuring instruments attached to the test specimen, the exciting forces of the oscillator can be controlled.

Model Tests:

In special cases the structural value of girders, riveted or welded, of various steels or alloys (Fig. 9) or of concrete can be compared simultaneously with one oscillator (5). Plotting frequency curves during the endurance test, certain changes in the dynamic properties of different materials or structures can be followed. Temperature measurements with thermocouples distributed over the test girders will indicate a weak spot where the first fracture or plastic flow will occur. Failure in any of the test girders makes evident the weakest structure or the weakest point which has to be improved or strengthened.

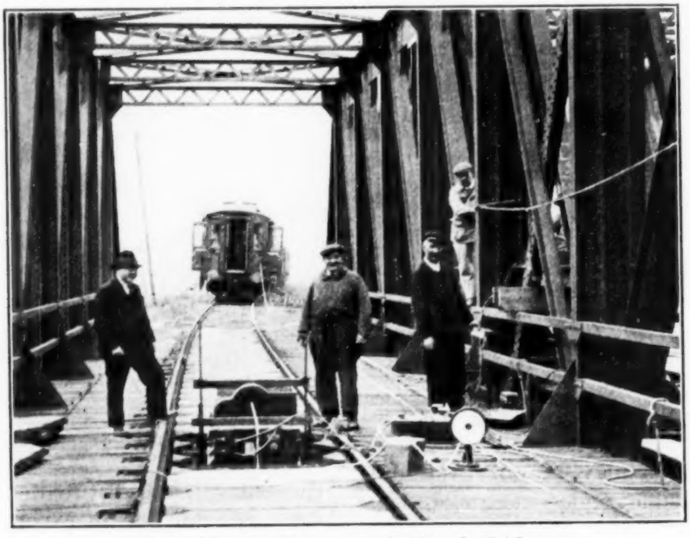


FIG. 10.—Oscillator on Railroad Bridge.

For a considerable time the need has been felt for machines capable of testing large size specimens in fatigue, particularly of such materials as concrete. The dynamic test presented in this paper meets this need and with relatively small oscillators, higher capacity and higher speeds can be obtained.

Field Tests:

Buildings.—Horizontal, vertical or torsional natural frequencies of ceilings, walls, towers, and foundations (Fig. 11) have been determined by field tests before or after the building is completed. Vibrations produced in a building by the machinery to be installed may become dangerous. Predetermination of this by experiments in the early stages of construction may save later expense, since small adjustment of masses, span length, deflection, or machine speed, made after study of oscillator test data, will help to avoid later difficulties.

Bridges.—The complex questions involved in impact stresses in bridges can be studied without using the usual live loads (6). It is sometimes difficult to use steam locomotives, which become all the more unsatisfactory due to their changing axle loads, caused by water and coal losses during the tests. To simulate the centrifugal forces of unbalanced steam locomotive driver axles, the oscillator has been placed on a platform car and pushed with a certain speed over the bridge. Damping capacities of various old and new highway and railroad structures have been determined by this method (5). The author developed nomographic charts for the determination of dynamic stress to be expected in railway bridges, due to steam locomotives (9).

Vertical, horizontal or torsional vibration has been applied to bridges by the arrangement shown in Fig. 10. In nearly all cases the connections will fail. This might be considered as another way to observe the effect of secondary stresses caused by the more or less stiff connections of the different members under reversal of loads.

Vehicles.—All sorts of vehicles have been studied in some measure. The damping qualities of a single spring or the entire spring suspension system of locomotives, passenger or freight cars or automobiles (Fig. 12) have been investigated for vertical, horizontal or torsional vibrations. Any sympathetic vibration between parts carried by the springs and the supporting structure (for example a bridge) has to be avoided as much as possible.

Aviation.—In the aviation field, the free vibration of a single propeller blade or crankshaft, as well as the dynamic behavior on the whole body (Fig. 13) has been investigated (7). One of the first failures of the "Zepplins" was caused by resonant vibration of the crankshaft of the engines.

Ships.—The following types of investigation are of definite significance to the Navy: Reproducing vibrations of the engines, the forced and natural vibrations of the complete hull, or of individual decks. Wherever resonant oscillations are found, structural changes can be made before the ship is completed. The damping effect of the water on a floating vessel (S. S. Bremen) has been measured; a specially constructed oscillator produced centrifugal forces up to 50,000 lb. at very low periods (2 revolutions per second).

Soil Investigation.—One of the newest developments in microseismic investigations is the dynamic soil tests (8, 11). The propagation of waves in the elastic soil in different strata give rise to particularly interesting geophysical phenomena such as diffraction, absorption, reflection, interference or superimposing

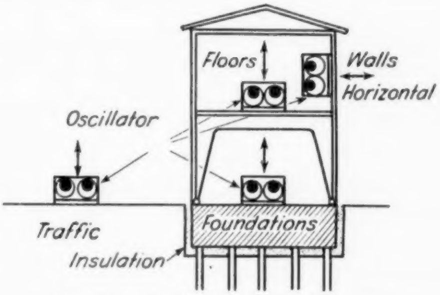


FIG. 11.—Oscillator in Buildings and Foundations.

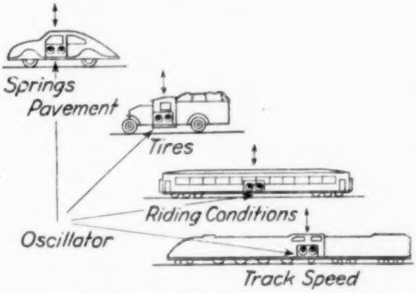


FIG. 12.—Oscillator in Vehicles.

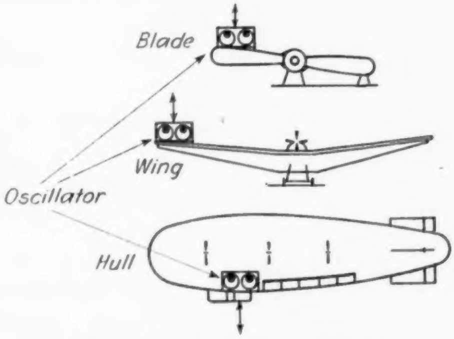


FIG. 13.—Oscillator in Aircraft.

effects, the discussion of which, however, falls without the scope of this paper. The propagation or phase speed of the mechanical waves in the subsoil depends to a certain amount upon its rigidity and hence its bearing power. A discontinuity in the rate of propagation indicates often an inclusion or interruption in the subsurface. In measuring the phase speed and vibration amplitudes, due to artificial oscillations, continuous diagrams of different strata, as well as of the bearing capacity of the subsoil have been drawn. For certain soils, slow speeds frequently have been found in earth fills (loose strata of less rigidity) and higher speeds in earth cuts (dense strata of greater rigidity). Such oscillator tests may take the place of borings. Borings at times are unreliable due to their arbitrary location. With artificial vibrations continuous cross-sections of undisturbed soil covering a complete area have been determined (8).

SUMMARY

The following table is an attempt to summarize some average values of dynamic properties for numerous vibrating systems investigated by the oscillator method (5):

System	Damping Angle, deg.	Tangent of Damping Angle	Logarithmic Decrement	Time to Build up Vibration, sec.	Amplifying Factor
Bridges, engine parts, ceilings, walls, towers, ship decks, masts, airplanes and airship parts	5.7	0.1	0.31	7.3	10
Foundation, ballast, buildings, springs of vehicles, street surfaces, weirs	11.3	0.2	0.62	3.7	5
Ships, certain soils, etc.	21.8	0.4	1.25	1.8	2.5

Advantages:

The advantages of the method are briefly:

1. An oscillator is less expensive than standard fatigue testing machine of equal capacity. Oscillators are easily operated both in the laboratory and in the field, and are independent of temperature, weather or other external conditions.

2. Many vibrating engineering systems, large or small can be tested with alternating centrifugal forces, which are independent of the force of gravity and therefore may be used in any direction.

3. Frequencies and centrifugal forces of the oscillators can be adjusted over a wide range (1 to 100 periods per sec. and 1 to 2,000,000 lb.) and can be made to simulate quite closely service conditions.

4. Dynamic loads can be combined with static loads, and fatigue and aging may be continued up to destruction of the complete system.

5. Comparisons between different materials and different constructions can be made simultaneously, making evident the better structure without further calculations.

6. The bearing capacity of soil strata may be determined in certain cases without resorting to borings.

Difficulties:

On the other hand, until greater experience has been had with oscillator tests success in all cases cannot be assured in advance. In certain cases, routine tests with standard testing machines will need to be made to check and supplement the oscillator tests.

When testing a vibrating system in the field, it may sometimes be difficult to avoid energy losses through radiation.

For very accurate tests a motor-generator set is required to control more closely the rotating speed of the revolving masses.

Acknowledgment.—The author wishes to express appreciation to Prof. G. E. Beggs, Dr. W. Spaeth and O. W. Grow for advice.

APPENDIX

THEORETICAL PRINCIPLES OF TESTING BY USE OF OSCILLATORS

The theory of the use of oscillators is covered quite thoroughly in the literature, as indicated in the bibliography.[4] Nearly every system in the engineering field is capable of vibrating, and may be set in motion by alternating forces of a sine form. Such alternating forces of a sine form can be readily produced by two revolving masses, eccentrically supported, and moving in opposite directions (Fig. 1). The two horizontal forces are neutralized automatically. The two remaining vertical forces have a sine form with an amplitude dependent on the eccentricity and a frequency equal to speed of rotation of the two masses. The correlation between amplitude A, speed V, acceleration a and time t is shown in Fig. 14, and can be explained in a simple way by one vector diagram (Fig. 15), the vector diagram demonstrating the difference in phase of 180 deg. between amplitude A, and acceleration (a) or the difference in phase of 90 deg. between amplitude and speed (ωA). The vibrating of a simple spring-supported mass by such an artificial force from low to higher frequencies will cause the amplitude of any oscillating system to increase up to the so-called resonance point and to diminish after the resonance peak is passed. In Fig. 16(a) the abscissa may represent the frequency of the impressed forces (n) or the cycles per second of the whole vibrating system; the ordinates represent the power (watts) necessary for revolving the two masses. The three curves shown are for varying damping capacities; the centrifugal forces rise as the square of the rotational speed of the eccentric masses. In Fig. 16(b), three different phase curves of the corresponding system are shown. In most cases these fundamental curves determine with sufficient accuracy certain dynamic properties of any vibrating system. By changing the relative position of the two revolving masses of the

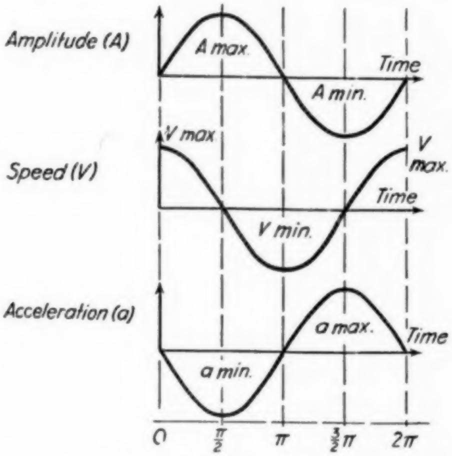


FIG. 14.—Correlation Between Time-Amplitude Time-Speed, and Time-Acceleration for One Period (5).

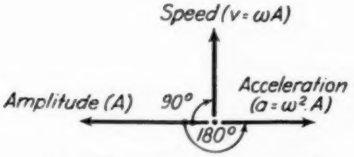
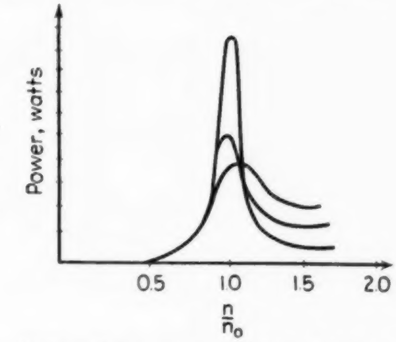
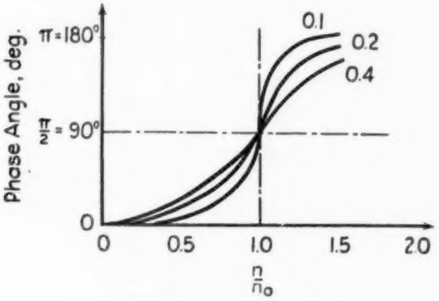


FIG. 15.—Vector Diagram (5).



(a) Power-frequency curve for forces, increasing with the square of the cycles (5).



(b) Phase-frequency curve (5).

FIG. 16.—Correlation Between Power and Phase Angle and the Ratio $\frac{n}{n_o} = \frac{\text{periods of the exciting force}}{\text{natural frequency}}$ for Three Different Damping Angles.

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[4] See p. 644.

oscillator, and by giving the two eccentric supports a difference in phase of 180 deg., the same tests can be carried out for torsional vibrations (Fig. 2(*b*)).

For normal tests, power-frequency curves are plotted (Fig. 3). The most important formulas for computing these curves are given below.

Characteristic Frequency:

1. For small damping values (most structural systems) the peak of the power-frequency curve gives the resonance frequency n_o with sufficient accuracy (Fig. 3).

2. Phase measurements are independent of the amplitude and are more accurate. The frequency (multiplied by 2π) for which the phase between the exciting force of the oscillator and the amplitude of the forced vibration system is 90 deg., also gives the resonance n_o.

Damping Values:

1. For small damping values, any line, *AB*, parallel to the no-load curve, *CD*, through the middle of the greatest amplitude *E* cuts the power frequency curve at the frequencies n_1 and n_2 (Fig. 3).

The damping angle is: $\tan \varphi = \frac{n_2 - n_1}{n_o}$

and the logarithmic decrement **(5)** is:

$$\delta = \pi \cdot \tan \varphi = \pi \cdot \frac{n_2 - n_1}{n_o} \quad \ldots\ldots\ldots (1)$$

where δ is a dimensionless unit.

2. The damping values with phase measurements may also be calculated in another way. As soon as the frequency is 0.62 of the characteristic frequency, n_o, the damping angle is equal to the phase angle between the exciting force (oscillator) and the amplitude (vibrating system)**(5)**. The damping angle is $\tan \varphi = \tan x$ for $n_x = 0.62\, n_o$

and the logarithmic decrement $\delta = \pi \tan x$ for

$$n_x = 0.62\, n_o \quad \ldots\ldots\ldots\ldots\ldots (2)$$

Amplifying factor:

The time required for any vibrating system to change from zero to maximum amplitudes (stable equilibrium) is infinite. To get a definite time value, it may be assumed that equilibrium is reached as soon as the amplitude has risen to 9/10 of its final value. The amplitude (A_{dyn}) is equal to the static amplitude (A_{stat}) multiplied by the amplifying factor *F*. The following equations may be used with sufficient accuracy:

$$A_{\text{dyn}} = F\, A_{\text{stat}} \quad \ldots\ldots\ldots\ldots\ldots (3)$$

$$F = \frac{\pi}{\delta} = \operatorname{cotan} \varphi \quad \ldots\ldots\ldots\ldots\ldots (4)$$

With these four very simple formulas routine calculations of quite a number of vibrating systems can be carried out with little mathematical difficulty and in most cases with sufficient accuracy.

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DISCUSSION

Mr. H. F. Moore.[1]—In this paper Mr. Bernhard has given a discussion of a mechanism for conducting repeated-stress tests which can be used both for testing specimens of metal, and for testing full-size structural parts, or even some full-size structures. This method—the use of the reversing action of a rotating unbalanced weight—has been used in former years, especially in England by Strohmeyer and I. H. Smith, and in this country by McAdam. However, Mr. Bernhard's paper suggests a much wider field for its use.

I should like to comment on the use of rise of temperature as an indication of approaching fatigue fracture. Tests in a number of laboratories have cast doubt on the reliability of this method. In the Illinois laboratory some specimens under reversed stress have heated up appreciably, and, without stopping the test or removing the load, the specimens have then cooled off and run for 100 million cycles without fracture. This action was unusual but occurred often enough to cause doubt as to the reliability of the rise of temperature test for determining endurance limit.

Mr. L. B. Tuckerman.[2]—The type of test for which Mr. Bernhard has developed this vibrator is one which will be used to an increasing extent in the study of structures. The apparatus which Mr. Bernhard has developed is one of the most useful forms for many purposes, and I do not want to give the impression that it will not prove very useful. However, I feel that Mr. Bernhard, like many enthusiasts, has somewhat slighted the difficulties of the problem and has rather overemphasized the advantages. Many of the statements he makes need some qualification. For example, he makes the statement that vibration tests on actual structures will result in a more complete analysis of the existing conditions, than can be obtained from tests in the laboratory. That is certainly true, but I doubt whether they will result in a more complete analysis unless we already have the results of tests on standard specimens in the laboratory, with which to compare them.

He says that the fundamental principle is simple. The fundamental principle is simple, but when you start to work out some of the equations you will realize that they are not quite so simple as the fundamental principles. He mentions only very briefly the fact that you may not have harmonic vibrations, and illustrates only one of the effects on the resonance curves, although vibrations which are not harmonic are far more common in structures than harmonic vibrations. First of all, the restoring forces in actual structures rarely are in proportion to the displacement; second, the dissipative forces are largely due to solid friction so that the damping is not proportional to the velocity. Consequently the very simple theory which he gives here is only a first approximation of what occurs in actual structures. It is ample for many rough approximations but if accurate

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[2] Assistant Chief, Division of Mechanics and Sound, National Bureau of Standards, Washington, D. C.

details are desired, one will find that that simple theory will not give the results you desire.

For instance, on this simple theory the vibrations of the structure will always have the frequency of the impressed forces, yet in a structure which I studied recently, we found that impulses with a frequency of 3200 per min. set up very strong and dangerous vibrations at a frequency of 1600 per min., and those in turn, set up very strong and dangerous vibrations at a frequency of 800 per min. That was a structure in which the restoring forces were not proportional to the displacement.

Curves such as he shows in Fig. 4 of the paper, in which the end of the resonance curve is tipped over on one side, are not so infrequent. They are characteristic of the familiar "hang-on" phenomena of structural vibrations. Vibrations once set up frequently refuse to stop even when the frequency is changed over a wide range. The importance of this paper warrants careful reading.

MR. W. P. ROOP.[3]—My thoughts are very similar to Mr. Tuckerman's, namely, that this is a type of test which is bound to be used increasingly. In addition to the points that he has mentioned, power absorption is something which has to be considered. We must watch the power leaks. These may occur at the most unexpected places. For example, one of the first things discovered when the attempt was made to induce vibrations on a ship in the water was that the main effect was the churning up of the water. That same aspect of power leaking off into unexpected directions may also prove very embarrassing on shore—when you find the pickle jars dropping off the shelf in the adjoining residence.

MR. L. J. LARSON.[4]—I should like to ask several questions. Do you propose to use this method to stress-relieve both welded and riveted structures? If the stresses in the structure are relieved by the application of loads which cause stresses, why will not the loads applied in service relieve the stresses in the same manner? What advantage is gained by subjecting the structure to a vibratory loading instead of service loads? From the curves shown, it appeared that up to a certain point the structure was improved by the vibratory loading, and after that the structure was damaged. Can the test be used to indicate when the structure begins to deteriorate, and is it proposed to test the structure periodically to determine when deterioration begins? What assurance is there that the test does not decrease the effective life of the structure?

MR. TUCKERMAN.—May I make one other comment? A rise in frequency of a structure does not necessarily mean that the structure is settling down and improving; nor does a lowering in frequency necessarily imply a weakening of the structure.

I have known of cases in which a rise in frequency was an indication of failure. In these cases, the vibrations finally loosened and detached some of the masses in the system which were previously being forced to move. With less mass in the vibrating system, the frequency was increased rather than lowered as failure began.

It is true that more usually lowering of frequency precedes failure because failure usually occurs where the elastic restraining forces act most strongly, but this is not necessarily so. Either a rise or a fall of frequency may be an indication of incipient failure.

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[4] Director of Welding Research, A. O. Smith Corp., Milwaukee, Wis.

MR. RUDOLF K. BERNHARD[5] (*author's closure*).—I want to thank the several discussers for their comments. The method of investigating specimens and structures by means of "induced vibration" is relatively new, and there is no doubt that there is still ample room for improving and supplementing this type of investigation.

In full agreement with H. F. Moore, referring to temperature increase, no attempt has yet been made to shorten the time required for determining the endurance limit on single small specimens. However, the increase in temperature when used as a means to indicate structural failures has been successfully applied in the case of complete riveted structures. The friction between various gusset plates causes, after a certain amount of vibration, as soon as the rivets start to loosen, a substantial rise in temperature—in certain cases more than 50 deg. Cent. above the temperature of the surrounding air. Such a temperature rise indicates the weakest spot in the structure several hours before the actual failure becomes visible, for example as a minute crack starting from a rivet hole and proceeding through the gusset plate.

An elaborate development of the exact theory, to which Mr. Tuckerman refers, could not be included in the compass of a short paper. The book on "Mechanical Vibration of Bridges," by E. Homann and the author, to which reference is made in the bibliography, gives a detailed development of the theory. The analysis of results is sometimes rather complex as indicated by Mr. Tuckerman. The following example is cited as a further illustration. Vibrating a steel truss in a vertical plane with an oscillator, three main typical frequencies might be excited: (1) The natural vertical frequency of a complete girder of medium span, the fundamental tone, between $\frac{1}{2}$ and 20 cycles per sec.; (2) the natural bending frequency of single members, chords, diagonals, etc., between 10 and 100 cycles per sec.; and (3) the natural torsional frequency of the free edge of an angle within the cross-section of a member between 50 and 300 cycles per sec. Keeping this in mind, the analysis can be substantially simplified. The results thus obtained have indicated that the assumption of a damping proportional to the speed is in most cases accurate enough for practical purposes. However the limitation in the application of the method, cited at the end of the paper, must be emphasized, namely, that the theory of resonance curves is only accurate enough for small damping values. Furthermore, it is true that a decrease in natural frequency may not always indicate a failure in the structure. Any failure in the foundations, especially in case the amount of vibrating soil adjacent to the foundation decreases, may cause an increase in frequency. Investigation of this type led to the discovery of undermined pillars caused by underwater currents.

Mr. Roop refers to a limitation of the method due to energy losses through the foundations. This of course might cause serious trouble in investigations of complete ships. However, in the structural field such energy losses through the foundations can in most cases be avoided by proper insulation. Furthermore, as in the case of the foundation cited above, this change in energy leakage has been used successfully to discover a serious failure in the foundation.

The four questions raised by Mr. Larson may be answered as follows:

1. The stress-relieving effect has been

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[5] Consulting Engineer, Baldwin-Southwark Corp., Philadelphia, Pa.

used on welded structures to get a more even distribution of peak stresses resulting from heat developed during the welding process.

2. By watching carefully any change in dynamic qualities (natural frequency, damping, amplification factor, etc.), i.e. plotting resonance curves, this stress-relieving effect can be followed, which cannot be done with service loads.

3. A decrease in natural frequency determined by periodic measurements of the dynamic qualities, both on welded and riveted structures, will indicate, with the exception of the above-mentioned case of change in energy leakage through the foundation, that the transition point, as shown in Fig. 6, has been passed. Hence, this will reveal that the retrogression period has started and reinforcement or complete renewal of the structure is required. A further change in the dynamic qualities will again indicate if the reinforcement has been effective, in most cases by a raised rigidity, hence, increase in natural frequency.

4. Investigations on actual structures should be made by all means so as not to decrease the full effective life of the building. During the investigations the excited amplitudes can always be kept very small, more than one-tenth of the amplitude which would induce the maximum allowable stresses. Hence, only a negligible part of the adjustment period (see Fig. 6) is used.

The author would like to see further investigations carried out by this method of "induced vibration," or other methods in order to gain more complete data on the special questions which have been raised by the discussers, some of which certainly need much more detailed studies. I believe that this method of "induced vibration" opens a field to the practical engineer to approach the difficult dynamic questions in a rather simple way. Practical tests of this type in cooperation with other methods and advanced theory will help to throw more light on the somewhat neglected "dynamic" study of our engineering structures and materials.

IMPROVEMENT IN THE ADAPTABILITY OF THE TUCKERMAN STRAIN GAGE

By L. A. Meisse[1]

Synopsis

Transfer mechanisms have been developed as described in this paper, for the purpose of improving the adaptability of the Tuckerman optical strain gage. These consist of a special construction of parallel flexure plates which transmit the movement from the specimens to the gage body. Some commercial applications of the system are given, and a broader use of similar flexure plate transfers to other types of strain gages is shown. The broadening of the adaptability of strain gages to general displacement measurements widens their scope of usefulness in the testing laboratory.

The principle of the cube corner reflection in an optical lever strain gage was discussed by Tuckerman in 1923.[2] The autocollimating feature together with the freedom from parallax effect has made the instruments admirably suited to applications beyond the scope of mechanical strain gages.

A number of improvements have been made in the construction of the instruments over the design originally described so that the present commercial instruments as shown in Figs. 1 and 2 are trustworthy to the highest degree. A notable improvement consists in embedding the roof angle prism in a metal cage with only the two aperture faces exposed. This makes a very solid mounting which will withstand considerable mechanical abuse without impairing the optical alignment. Notable also is the self-aligning spring support for the lozenge. The lozenge is now made from optically polished stellite constituting the third reflecting face instead of the separate right-angle prism mounted on a steel lozenge formerly used.

The preferred commercial size for these gage bodies is 1-in. gage length, larger effective gage lengths being obtained with extension fingers. The Tuckerman gages at present commercially available can be depended upon for readings of 0.000005 in.

For measurements on flat or slightly curved faces the lozenge contacting with the specimen is adequate. In cases of inaccessibility or in case of obstructions between the gage marks these instruments have not been applicable.

The system originated by Tuckerman can be greatly extended by transfer mechanisms without the construction of special instruments for each individual application. This has the advantage that a large variety of measurements can be made with relatively little expenditure beyond the acquisition of a standard gage, obviating the necessity of make-

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[1] Research Director, The Ohio Brass Co., Mansfield, Ohio.

[2] L. B. Tuckerman, "Optical Strain Gages and Extensometers," *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part II, p. 602 (1923).

shift setups which would impair the reliability of the test.

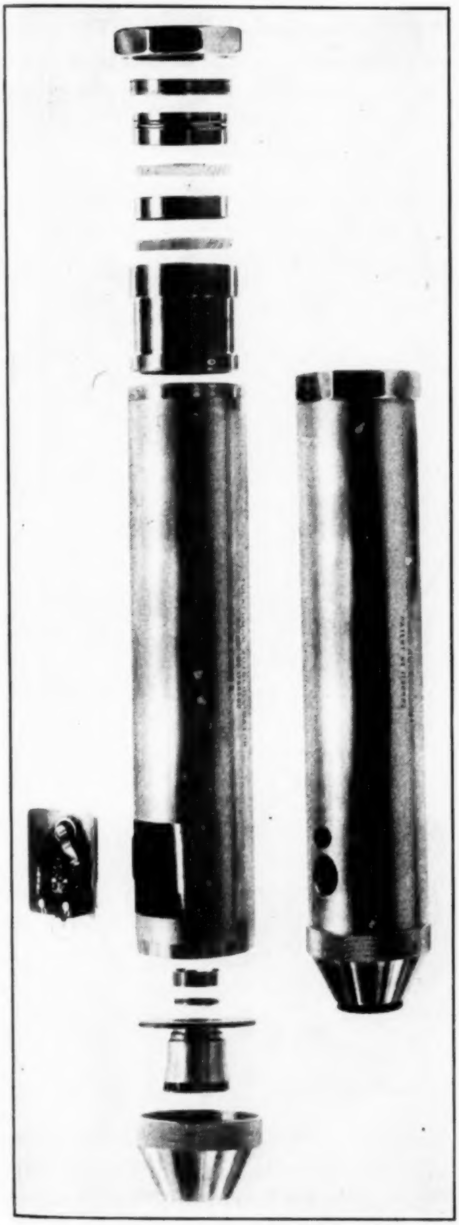

FIG. 1.—Tuckerman Auto-Collimator.

These transfer mechanisms consist essentially of a set of parallel flexure plates, which transmit the movement from the gage points rigidly and without distortion to the Martens lozenge. For the sake of ridigity and unaltered transposition ratio, and above all for simplicity of manufacture, these transfer devices were machined from a single piece of material.

Since the transformation ratio is unaffected by the thermal expansion of the material, the transfer mechanisms do not affect the temperature characteristic of the optics. Steel, invar and duraluminum have been used at various times for the manufacture of the instruments, the choice depending solely on weight and corrosion requirements.

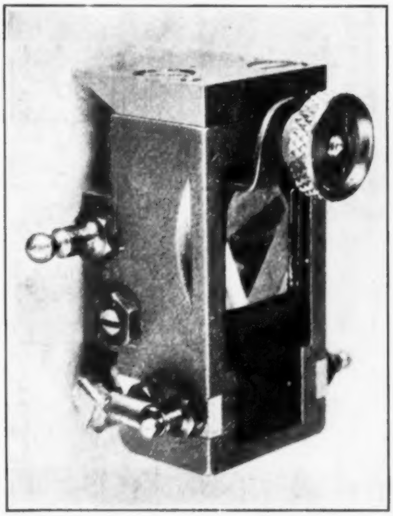

FIG. 2.—Tuckerman Strain Gage.

Construction:

The design in its most useful form is given in Fig. 3. It comprizes two angular rigid frames *a* and *b* connected by two parallel rigid spacers *c* and *d* of equal length connected to the frames by delicate flexing sections as shown. The standard commercial gage body of the Tuckerman gage *f* is shown in position.

The engagement points are reversible to change the gage length from 1 in. to 2 in.

To make the unit adaptable to the various types of measurements which come up in a laboratory, it is necessary to attach various kinds of engagement points. Several examples are shown in Fig. 4, in which *A* represents engagement points for the measurement of internal distances, such as hole distortions; *B* is intended for height measurements or movement of a point normal to a plane; and *C* shows an extension comprising a tube and feeler rod to reach inaccessible places. The design *A* was used in the test shown in Fig. 5 and designs *B* and *C* in the test shown in Fig. 6. The tubular extension of *C* was in this latter case fitted through the suspension pin of the insulator in order to measure displacements between the pin and the porcelain dielectric in the interior of the insulator.

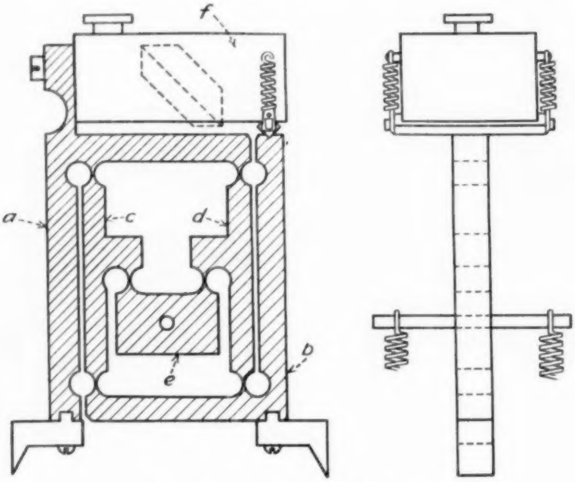

FIG. 3.—Transfer Mechanism.

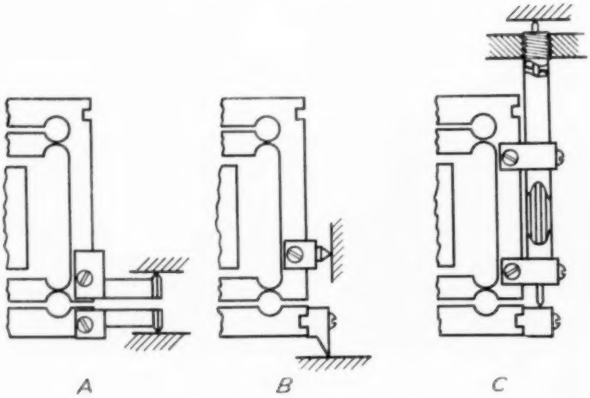

FIG. 4.—Showing Various Engagement Points.

Engagement Pressure.—Due to the inherent restraining action of the flexure plates a larger contact pressure is necessary than is required with the standard

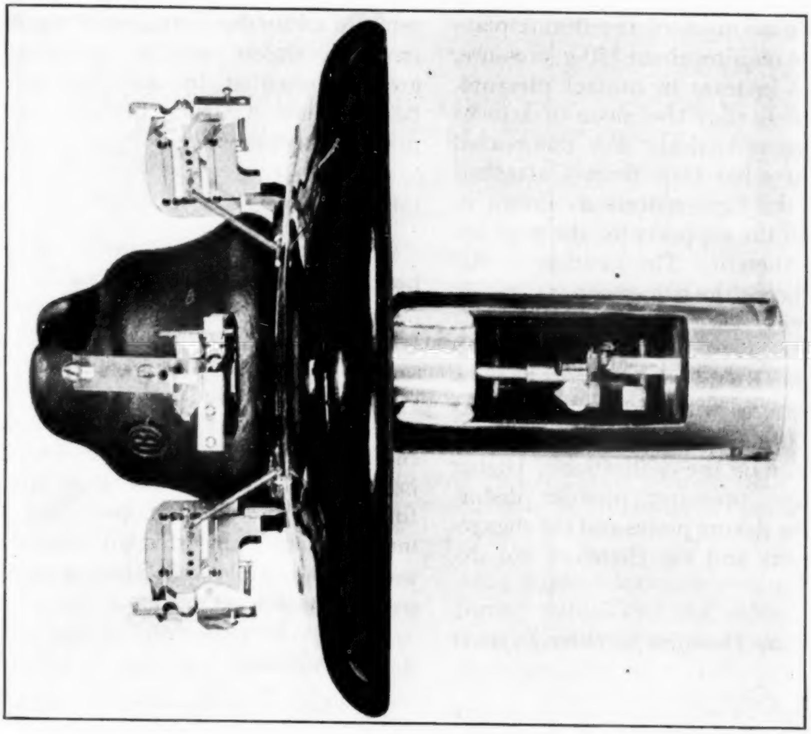

FIG. 6.—Measurements on a Typical High-Tension Suspension Insulator for the Displacement and Stresses in the Hardware.

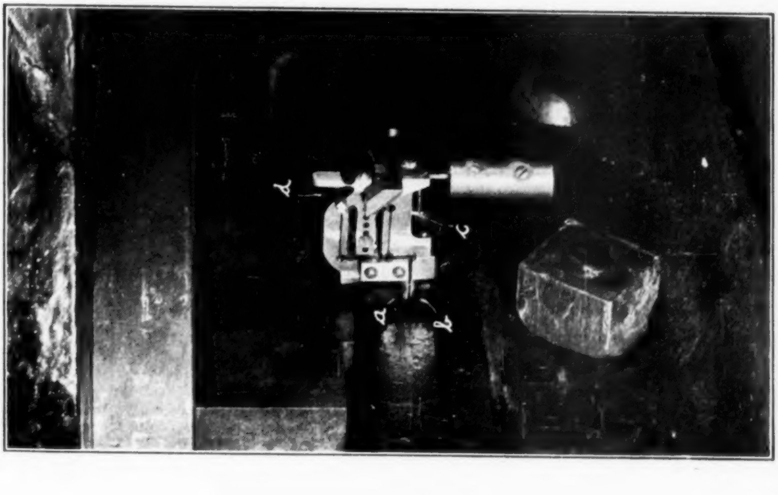

FIG. 5.—Measurement of the Hole Distortion in the Head of a Rail Due to Car-Wheel Pressure.

Tuckerman gage. While 20-g. contact pressure is generally sufficient for the latter, the addition of the flexure-plate extensions requires about 150-g. pressure. With this increase in contact pressure, it is obvious that the mode of attachment is more critical. For this reason a crossbar *e* has been flexibly attached between the rigid spacers as shown in Fig. 3 and the supports for the gage are attached thereto. The location of this crossbar keeps the suspension invariably at midpoint between the engagement points irrespective of the gage length extension. By this arrangement engagement pressures within reasonable limits (up to 300 g.) have been found to have no influence on the calibration. Higher engagement pressures produce distortions in the flexure plates and the engagement points and are therefore not desirable.

Flexure Plate Transfers for Other Types of Gages:

Obviously these same types of flexure plate transfer mechanisms can be fitted to other types of laboratory gages with a view to increasing their adaptability. The single or double Martens mirror, which is a forerunner of the Tuckerman gage, can be attached to this transfer mechanism and thereby improve the performance. Of course the limitations of a single reflection set an indefinite limit to the magnification usable on account of the inherent uncertainty of the readings. Huggenberger extensometers and all similar instruments requiring mounting closely onto the specimen are greatly benefited by addition of the flexure plate transfer devices of the principle here described.

Conclusion:

The flexure plate transfer devices herein shown have made possible precision measurements in inaccessible places which otherwise would have required the construction of special gages of very limited range of usefulness. The Tuckerman optical lever instruments, if coupled with these transfer devices, can be extended into a broad-range laboratory tool, and it is hoped that the methods herein suggested will expand the use of the triple reflection, autocollimated optical lever system.

Acknowledgments:

The author is indebted to the following for their interest and support: L. B. Tuckerman, Assistant Chief, Division of Mechanics and Sound, National Bureau of Standards; Commander C. R. Gayhart, U. S. Navy Dept., Washington, D. C.; The American Instrument Co.; and the many associates at The Ohio Brass Co. in particular the author's former private assistant Harold E. Swigart, for their loyal help.

DETERMINATION OF THE EDGE CORRECTION IN THE MEASUREMENT OF DIELECTRIC CONSTANT[1]

By Arnold H. Scott[2]

Synopsis

Nine methods of determining the dielectric constants of sheet insulating materials were used in making measurements on the same specimens with seven arrangements of circular electrodes. Two methods of computation were used with two of the arrangements. The values obtained with guard-ring electrodes were assumed to be correct. The errors of the other methods were obtained by a comparison with the guard-ring values. Two of the methods gave values which were in error by less than 1 per cent. Three other methods gave values which were in error by less than 2 per cent while the values obtained by the other three methods were in error by 2½ to 7 per cent.

Last year a paper was presented before the Society by Curtis and Scott[3] giving a theoretical discussion of the determination of the edge correction for ten different arrangements of plate electrodes when used in the determination of the dielectric constants of materials in sheet form. Six of these arrangements were concerned with circular electrodes, the remainder with narrow strips. The formulas given for these various cases were considered from the standpoint of derivation and applicability. It was predicted that some of the formulas would give values which had appreciable errors while others would give reliable values.

The present paper presents results of electrical measurements made to check the accuracy of the formulas for edge correction of circular electrodes. The values for dielectric constant obtained using guard electrodes were taken to be correct values and the values obtained from other arrangements of electrodes were compared with them.

The specimens on which measurements were made were of glass, hard rubber, white marble, and blue marble. They were cut from uniform sheets of material in the form of circular disks about 15 cm. in diameter. The glass specimens were cut from a piece of plate glass about 4.6 mm. thick, no care being taken in its selection other than the uniformity in thickness. The hard rubber specimens were cut from a piece of hard rubber about 4.7 mm. thick obtained from the general supply. The marble specimens were cut from slabs which were carefully selected as to uniformity of appearance. They varied in thickness from 6.7 to 7.7 mm. Four specimens from each material were prepared.

Arrangements of Electrodes

Tinfoil was used as the electrode material. This was applied with a thin

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[1] Publication approved by the Director of the National Bureau of Standards of the U. S. Department of Commerce.

[2] Assistant Physicist, National Bureau of Standards, Washington, D. C.

[3] Harvey L. Curtis and Arnold H. Scott, "Edge Correction in Determination of Dielectric Constant," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 815 (1936).

coat of petrolatum and carefully rolled down on the surface of the specimen with a narrow roller until no visible imprint could be made on the tinfoil with the roller. Experiments were made which showed that this procedure of rolling was necessary if the formation of bubbles under the electrodes was to be avoided. When the tinfoil was rubbed down in the usual manner, gas bubbles became visible under the tinfoil in less than 24 hr. When, however, the tinfoil was rolled down with a narrow roller on which considerable pressure was exerted until no imprint could be made on the tinfoil, no bubbles appeared under the tinfoil within several months.

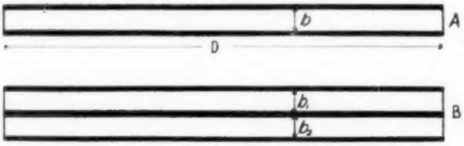

FIG. 1.—Electrode Arrangements Where the Electrodes Extend to the Edges of the Specimens

For arrangement *A* the dielectric constant was computed by means of the equation:

$$K = \frac{16bC}{1.113D^2} - \frac{2b}{\pi D}\left[\ln\frac{8\pi D}{b} - 3\right]$$

where C = capacitance,
K = dielectric constant, and
$\ln$ = natural logarithm.

Arrangement *B* was obtained by facing together two specimens having electrode arrangements shown in *A*. The dielectric constant was computed by means of the equation:

$$K = \frac{16b_1b_2}{1.113\,(b_1 + b_2)D^2}\left[C - \frac{0.6165D}{\pi}\right]$$

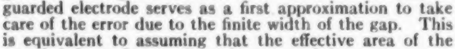

FIG. 2.—Electrode Arrangement When a Guard Electrode Is Used.

The dielectric constant was computed by means of the equation:

$$K = \frac{16bC}{1.113\,(D + y)^2}$$

The addition of the width of the gap, y, between the guarded and guard electrodes to the diameter of the guarded electrode serves as a first approximation to take care of the error due to the finite width of the gap. This is equivalent to assuming that the effective area of the guarded electrode extends to the center of the gap.

The electrodes were applied to the specimen only once and served for all the various arrangements. They were cut and modified as needed for the different arrangements but were not removed and replaced. This was accomplished by using the arrangements for the largest electrodes first. The tinfoil was applied over the entire surface of each side of the specimen as shown in Fig. 1. When measurements had been completed on these arrangements one of the electrodes of each specimen was cut with a circular cutter so that a very narrow ring was removed leaving a small electrode surrounded by a guard electrode as shown in Fig. 2. After measurements had been made with this arrangement, the guard electrode was removed as shown in Fig. 3. Following measurements with these arrangements the large electrode was cut and the outer piece removed so that two electrodes of the same size were left

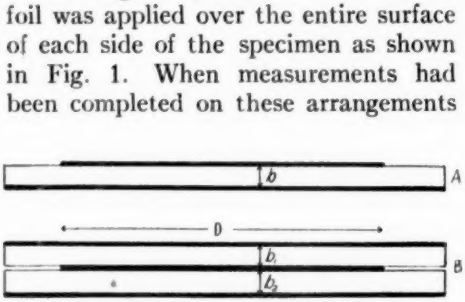

FIG. 3.—Electrode Arrangements When One Electrode Extends to the Edge of the Specimen and a Smaller Electrode Is Centered on the Opposite Face.

Two methods of computing the dielectric constant were used with arrangement *A*. The equations used were:

$$K = \frac{C}{1.113\left[\frac{D^2}{16b} + \frac{D}{4\pi}\left(\ln\frac{4\pi D}{b} - 3\right)\right]}$$

and

$$K = \frac{16b\,(C - E)}{1.113D^2}$$

The value of E was obtained from a curve published by Hoch.

Arrangement *B* was obtained by facing together two specimens having electrode arrangements shown in *A*. The dielectric constant was computed by means of the equation

$$K = \frac{C}{1.113\left[\frac{D^2\,(b_1 + b_2)}{16b_1b_2} + \frac{0.6932D}{\pi}\right]}$$

centered on opposite sides of the specimen as shown in Fig. 4.

The equations for circular electrodes given in last year's paper[3] were used for the computations of the dielectric constants. These equations are given in the captions under the figures. The correction terms for electrode thickness are omitted from these equations because the tinfoil electrodes were only 0.003 cm. thick and the correction for this thickness was negligible. In these equations the capacitances are given in micromicrofarads and the dimensions of the specimens in centimeters. The logarithm used is the natural logarithm.

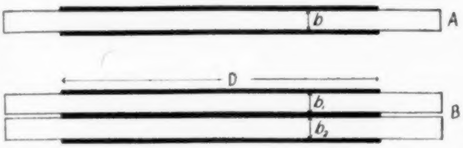

FIG. 4.—Electrode Arrangements Where the Electrodes Are of Equal Diameter but Smaller than the Specimen.

Two methods of computing the dielectric constant were used with arrangement A. The equations used were:

$$K = \frac{C}{1.113\left[\frac{D^2}{16b} + \frac{D}{8\pi}\left(ln\frac{8\pi D}{b} - 3\right)\right]}$$

and

$$K = \frac{16b\,(C - E)}{1.113D^2}$$

The value of E was obtained from a curve published by Hoch.

Arrangement B was obtained by facing together two specimens having electrode arrangements shown in A. The dielectric constant was computed by means of the equation

$$K = \frac{C}{1.113\left[\frac{D^2\,(b_1 + b_2)}{16(b_1 b_2)} + \frac{0.5539D}{\pi}\right]}$$

For two of the arrangements, Fig. 3A and Fig. 4A, the dielectric constants were also computed by a method described by Hoch.[4] The value of the edge capacitance, E, was taken as 11/11.43 of the value given by the curve published by Hoch since his curve was given for specimens having electrodes 4½ in. (11.43 cm.) in diameter while the electrodes used in the present investigation were 11.0 cm. in diameter. This assumes that within the required accuracy the edge-correction capacitance is proportional to the circumference, which is a plausible assumption for small changes in diameter.

ELECTRICAL MEASUREMENTS

The capacitance measurements were made with a conjugate Schering bridge provided with an earthing arm.[5] The bridge was well shielded and was operated inside an earthed cage. A balance of the bridge was first obtained without the specimen being connected to it. Then the specimen was connected in parallel with the standard capacitor and the bridge rebalanced. The capacitance of the specimen was taken as equal to the change in capacitance of the standard capacitor necessary to bring the bridge again into balance.

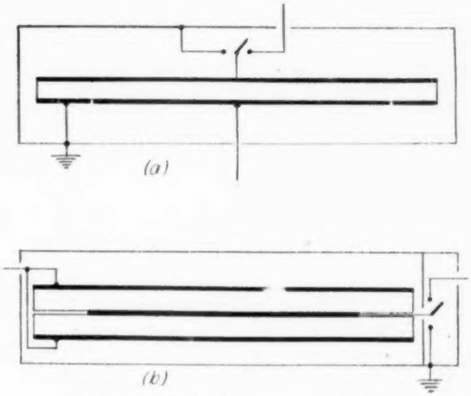

FIG. 5.—Shielding Arrangements.

(a) For a single specimen.
(b) For a pair of specimens.

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[4] E. T. Hoch, "Electrode Effects in Measurement of Power Factor and Dielectric Constant of Sheet Insulating Materials," *Bell System Technical Journal*, Vol. 5, p. 555 (1926).

[5] This bridge is the same as the one described in Tentative Methods of Testing Electrical Insulating Material for Power Factor and Dielectric Constant (D 150 – 36 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 955 (1936); also 1936 Book of A.S.T.M. Tentative Standards, p. 951.

The source of the 1000 cycles per sec. measuring current was a motor-generator set rur from storage batteries. A potential of 110 v. was applied across the bridge.

The specimens were placed in a metal box which served as a shield for the specimen and was part of a constant temperature chamber. When measured singly the specimens were mounted as shown diagrammatically in Fig. 5 (*a*). The switch used for disconnecting the high-potential lead of the specimen from the bridge and connecting it to earth was mounted in the box. This served effectively to shield the switch, thus eliminating capacitance error in the switch. When measured in pairs the specimens were mounted as shown diagrammatically in Fig. 5 (*b*). Here it was necessary to mount the switch in a separate compartment. The shielding in both cases was designed to eliminate as far as possible direct capacitance between the leads and between each lead and the opposite electrode. This effectively removed from the measurement any capacitance other than the capacitance between the electrodes in all cases except one. When the specimens were measured in pairs and the center electrode was smaller than the outer electrode, it was necessary to make connection to the center electrode with a small wire. This wire could not be shielded along the distance from the edge of the specimens to the center electrode so that the capacitance of this lead to the outer electrode was included in the measured capacitance.

TABLE I.—DIELECTRIC CONSTANTS.
Average of four determinations.

	Electrode Arrangement								
	2	1*A*	1*B*	3*A*	3*A*[a]	3*B*	4*A*	4*A*[a]	4*B*
Glass	7.76	7.78	7.79	7.40	7.74	7.80	7.35	7.73	7.58
Hard rubber	3.12	3.07	3.10	3.04	3.07	3.13	2.98	3.08	3.03
White marble	8.60	8.57	8.58	8.24	8.53	8.68	8.02	8.53	8.28
Blue marble	8.75	8.71	8.73	8.38	8.72	8.83	8.18	8.69	8.45

[a] Computed by method given by Hoch.

TABLE II.—PERCENTAGE DIFFERENCE BETWEEN COMPUTED DIELECTRIC CONSTANT AND GUARD ELECTRODE VALUE.

	Electrode Arrangement							
	1*A*	1*B*	3*A*	3*A*[a]	3*B*	4*A*	4*A*[a]	4 *B*
Glass	+0.3	+0.4	−4.6	−0.2	+0.5	−5.3	−0.4	−2.3
Hard rubber	−1.6	−0.6	−2.5	−1.6	+0.3	−4.4	−1.3	−2.9
White marble	−0.4	−0.2	−4.2	−0.8	+0.9	−6.7	−0.8	−3.7
Blue marble	−0.5	−0.2	−4.2	−0.3	+0.9	−6.5	−0.7	−3.4
Average	−0.6	−0.2	−3.9	−0.7	+0.6	−5.7	−0.8	−3.1

[a] Computed by method given by Hoch.

The specimens were maintained at 30 C. in a dry atmosphere for at least 48 hr. before measurements were made. The capacitance was measured without removing the specimens from the chamber. The marble specimens were dried by placing them in an oven at 115 C. for 24 hr. before being placed in a measuring chamber.

RESULTS

The values of the dielectric constants as obtained by the different methods and arrangements of electrodes are given in Table I. Each figure is the average of the determinations made on the four specimens of each material. Assuming the guard-electrode values to be correct the percentage errors of the determinations made by the other methods were computed. These percentage errors are given in Table II.

It was predicted in last year's paper[3] that arrangements 1*A* and 1*B* would give values which were too high, that arrangements 3*A*, 4*A* and 4*B* would give values too low and that arrangement 3*B* would give values that were very nearly correct when the equations given for each were used in the computations. These predictions were correct for all the arrangements except 1*A* and 1*B*. The values obtained with these arrangements were slightly too small rather than too large, as predicted, except for glass. The higher values for glass are probably accounted for by the fact that the glass specimens were slightly beveled at the edge. This was done by the glass worker to take off the sharp edge. Thus the specimen extended slightly beyond the edge of the electrodes. This gave rise to edge capacitances larger than contemplated by the equations and gave errors in computation which made the values too large. Presumably, then, these arrangements would have given values for glass too small, in line with the values for the other materials if the glass had not extended beyond the edge of the electrodes.

It can be concluded, then, that arrangements 1*A* and 1*B* give values of dielectric constant that are slightly too small, the percentage error being 0.5 per cent or less for all the materials except hard rubber for which the error was as much as 1.6 per cent. This would indicate that the errors in these arrangements are greater for materials of low dielectric constant.

Arrangements 3*A*, 4*A* and 4*B* yield results that are too small by several per cent, the average errors being 3.9, 5.7, and 3.1 per cent, respectively. If, however, Hoch's method of computation is used for arrangements 3*A* and 4*A* the values obtained are much more accurate. Here again the error is greatest for the specimens having the lowest dielectric constant.

Arrangement 3*B* yielded values which were all too high but the errors were less than 1 per cent. It was in this arrangement that the lead to the center electrode could not be shielded for the distance of about 2 cm. where it passed between the edges of the specimens. There was appreciable capacitance between this portion of the lead and the outer electrode and the equation does not allow for this. If this capacitance had been determined and due allowance made for it, the values of the dielectric constant would have been lowered and the error decreased.

To determine the error caused by the capacitance of the lead, the dielectric constant of fourteen pairs of marble specimens was measured using dummy leads. The dummy leads were made as near as possible like the real leads. The capacitance of the test unit was measured with the dummy lead in place and again after the dummy lead had been removed. The capacitance of the dummy lead and hence of the real lead was the difference between the two measured capacitances. The average error in the dielectric constant of the marble specimens when no correction was made for the lead capacitance was 0.9 per cent. The average error when correction was made for the lead capacitance was 0.4 per cent. The lead capacitance thus caused an error of about 0.5 per cent.

The amount of the edge correction is much greater in some arrangements than in others. If the correction terms were all ignored in the various equations used in the computations of the dielectric constants, the values for the various arrangements of electrodes would be in error by the percentages shown in Table III. Arrangements 1*A* and 1*B* would have given errors of only about 1 per cent by this method. The other arrangements would have given much larger errors. Arrangements 3*A* and 3*B* would have given a value as much as 13 per cent too high if no correction had been made for the edge capacitance.

TABLE III.—PERCENTAGE ERROR IN UNCORRECTED VALUES OF K.

	Electrode Arrangement					
	1*A*	1*B*	3*A*	3*B*	4*A*	4*B*
Glass	1.1	1.0	9.2	8.0	3.3	3.5
Hard rubber	1.0	0.6	11.8	7.7	4.2	2.9
White marble	0.7	0.6	13.1	13.3	4.7	5.7
Blue marble	0.7	0.6	13.5	12.3	4.8	5.5

According to these results, dielectric constants obtained by means of arrangements 1*B* and 3*B*, correction being made for lead capacitance, will be in error by less than 1 per cent. Dielectric constants having errors less than 2 per cent can be obtained from arrangements 1*A* and 1*B* using no corrections and from arrangements 3*A* and 4*A* using Hoch's method of computation. The other methods, however, will give results having errors of 2.5 to 7 per cent.

MEASURING ELASTIC DRIFT

By Robert W. Carson[1]

Synopsis

Spring elements in precision instruments may be subject to appreciable errors as the result of elastic drift, or deviations in elastic deflections occurring with continued time under a constant load. A recording electronic micrometer, sensitive to deviations as small as a millionth of an inch, was developed to measure deflections of spring members without disturbing the load deflection. Typical records show that drift continues indefinitely at a decreasing rate as long as the load remains. Test procedure is described for drift measurements on flat strip specimens and actual elastic elements used in instruments.

Since the elastic deflection of metal is widely used in precision instruments as a measurement of force, the ideal elastic material would have deflections exactly proportional to force under all conditions. Deviations from proportionality are caused by temperature, stress intensity, elastic hysteresis, and also by elastic drift during the time that the load is maintained. Elastic drift, or the deviations caused by time under load, have been difficult to measure with precision, although serious errors in instrument performance may result from this effect (**1**)[2]. More effective control of drift has been difficult because of the problem of determining the factors affecting this deviation, particularly as these factors apply to actual elastic elements used in precision instruments where stresses are much lower than the conventional elastic limit.

Elastic drift is a change in the deflection of a stressed member under a constant load, a change occurring only with the passage of time. This effect has been variously designated as creep, drift, elastic lag, temporary set, elastic fatigue, zero shift, elastic after-working, aging and mechanical hysteresis. One difficulty in establishing a term such as "elastic drift" for this deviation is that its effect on instrument performance often is difficult to distinguish from elastic hysteresis or the energy lost in rapidly carrying a material through a cycle of stresses. This energy loss is practically independent of time provided the cycle is carried through rapidly. If the cycle is carried out slowly, then the elastic deviation will be a combination of elastic hysteresis and elastic drift. With a steady load, however, the deviations occurring with continued time under load are unaffected by elastic hysteresis. In many respects, elastic drift is but a continuation of high-temperature creep into lower normal temperature conditions. Creep is perhaps a better term than drift for this effect, but it seems that creep is now generally associated

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[1] Instrument Specialties Co., Little Falls, N. J.

[2] The boldface numbers in parentheses refer to the reports and papers given in the bibliography appended to this paper, see p. 668.

with effects observed under high-temperature conditions. Since the deviations considered here occur at normal temperatures the term "elastic drift" is used.

Regardless of how it is designated by various investigators, the effect is the same. Under a constant load, the deflection slowly increases; after removing the load some of the deflection remains for a time. Under load, the drift or increase in deflection continues at a decreasing rate; after removing the load, the small residual deflection slowly decreases and may eventually disappear.

Elastic drift has been measured by both dynamic (2) and static (3) methods. By observing the decrement of a long-period torsional pendulum, the combined effect of drift and elastic hysteresis has been measured under conditions of alternate reversal of stress. In most instrument applications, however, the elastic elements are subject to a stress of varying intensity in but one direction. Under such conditions elastic drift is usually of greater significance than hysteresis. Static tests therefore give data that are more readily applied to conditions found in precision instruments. However, static tests not only require extremely sensitive measuring methods, but the measuring method used must not affect the small deviations being measured.

Although drift effects may be large enough to present a serious problem in precision elastic members, the deviations are of such small magnitude that accurate direct measurements are difficult to make by usual means. For example, an altimeter diaphragm may have a total deflection of 0.100 in., and good performance may require drift to be less than 0.1 per cent of the load deflection during an hour under maximum load. The total deviation during the hour therefore will be no greater than 0.0001 in., and to measure the rate at which this deviation occurs requires a measuring means sensitive to displacements of a few millionths of an inch. Of course, during the test all outside disturbances, temperature changes and variations in load must be so closely controlled that they do not affect the measured deviations. Then also, the measuring system itself must not affect the deflection of the specimen. The problem therefore is to measure small deviations with a high sensitivity without placing any load on the specimen, and to maintain test conditions constant during the test period.

LIMITATIONS OF USUAL MEASURING METHODS

Tests on elastic elements have been made with fair accuracy by placing them under load for extended periods of time. Since drift increases with time, a long-time test may give a total deviation large enough for comparative purposes, but there is always the possibility that some disturbing condition may have occurred during the test period. Tests on wire materials have been made using long gage lengths in order to obtain a higher degree of sensitivity, and optical methods of measuring deviations can, of course, be applied to usual instrument elements. Neither of these methods, however, gave the desired results on small specimens or elastic elements within the desired time limitations.

Early attempts to make short-time determinations on actual instrument spring elements using optical methods were not entirely successful, and the equipment needed to obtain a running record of deviations was too complicated. In making direct observations, a large number of readings were necessary, particularly at the start of the test when the drift was relatively rapid. Interferometer methods were not successful because of the difficulty in eliminating errors caused by the load imposed on the test specimen, and mechanical means of magnification were found to have the same limitations. The use of a micrometer screw, as shown in Fig. 1, with electrical means for indicating point of contact between the micrometer tip and specimen was not successful in early attempts because of the appreciable and variable contact pressure required. Arcing quickly destroyed sharp-pointed contacts, and with broad contact faces electrostatic attraction affected the deflection of the specimen under test.

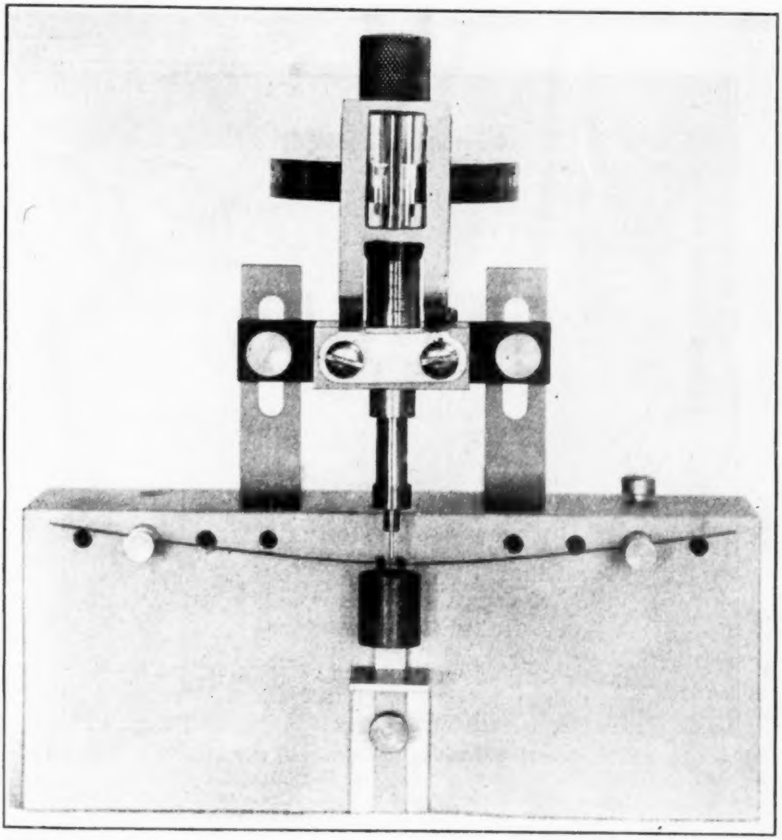

FIG. 1.—With Electrical Indication of Micrometer Contact, Pitting of the Sharp Contact Point Caused Relatively Large Errors in Measurement of the Load Deflection.

Contact difficulties were finally eliminated by using electronic means for indicating the instant of contact between the micrometer tip and the specimen. With a low-voltage contact circuit, a sharp contact point eliminated electrostatic attraction. A contact circuit of 50 megohms resistance reduced currents enough to prevent pitting of the contact point. This electronic indicator for determining instant of contact was found to operate for displacements of less than a millionth of an inch. Since such small displacements could not be read on the micrometer or on a large-diameter drum on the micrometer barrel, micrometer rotation was magnified mechanically by a constant tension wire and pulley drive, and the magnified movement was then recorded on a paper chart. For the maximum sensitivity so far required in drift tests, the mechanical magnification is arranged so that a 6-in. movement of the recording pen across the chart represented a displacement of nearly 0.005 in. under the micrometer tip. A millionth of an inch therefore is approximately 0.01 in. on the chart.

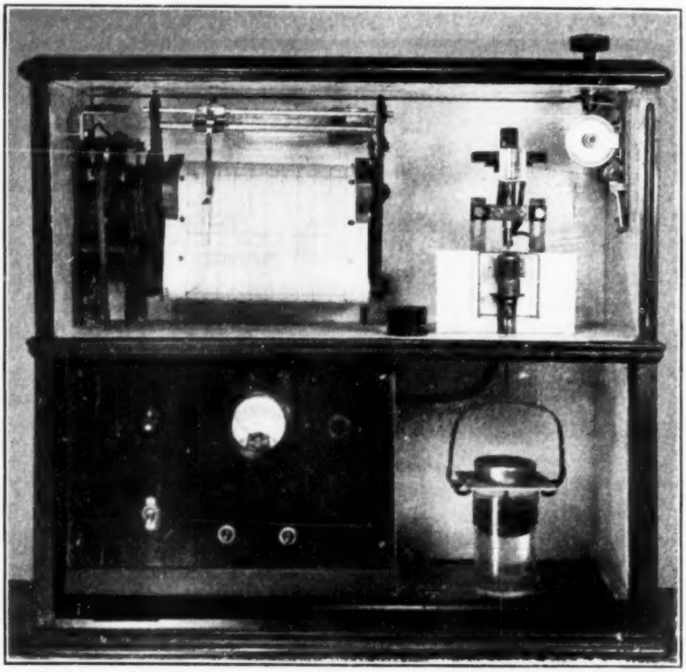

FIG. 2.—The Electronic Micrometer Sensitive to Deviations of a Millionth of an Inch in Elastic Deflections.

Contact with the metal bellows under test is indicated by the instrument on the panel. A follow-up mechanism keeps the micrometer just touching the specimen, and records the deviations on the clock-driven paper chart.

SELF RECORDING FOR GREATER ACCURACY

In order to eliminate human errors in setting the micrometer and to reduce disturbances that result from bodily contact with the instrument, the micrometer was automatically brought just to the point of contact by a small clock motor with a 1-r.p.m. output shaft for the pen drive. The electronic indicator through a relay controlled the current to the clock motor so as to stop the motor at the instant of contact when the micrometer tip was brought down against the specimen. As drift increased, the motor drive thus provided an automatic follow-up motion for the micrometer, keeping it always just touching the specimen. Another clock motor advanced the paper chart, thereby giving a continuous automatic record of increasing deflections or drift under load with the elimination of human errors in taking readings.

Since only a small amplitude of vibration might bring the specimen in contact with the micrometer tip and cause an automatic advancement of the micrometer, it has been found desirable to eliminate all outside disturbances as much as possible. Vibrations are isolated by mounting the micrometer on a foundation independent of the building structure, in a location free from traffic disturbances The relay controlling the micrometer drive motor is mounted separately from the micrometer case so that its operation does not disturb the specimen. A glass inclosure for the micrometer shields it from air currents, and absorbs some of the radiant heat that might otherwise change the temperature of the micrometer parts. A dashpot for the load is used to damp out both horizontal and vertical movements of the load. These details are shown in Fig. 2, a photograph of the complete instrument in one of its early forms.

Check tests have demonstrated that the electronic micrometer will repeat within a millionth of an inch, and that the sensitivity of the electronic contact indicator is something less than a millionth of an inch. The limit of sensitivity has not been determined since the present sensitivity is sufficient to measure accurately the hysteresis in a specimen under low stress during load periods lasting no longer than 10 to 30 min. This short time minimizes the possibility of errors caused by outside disturbances and temperature changes.

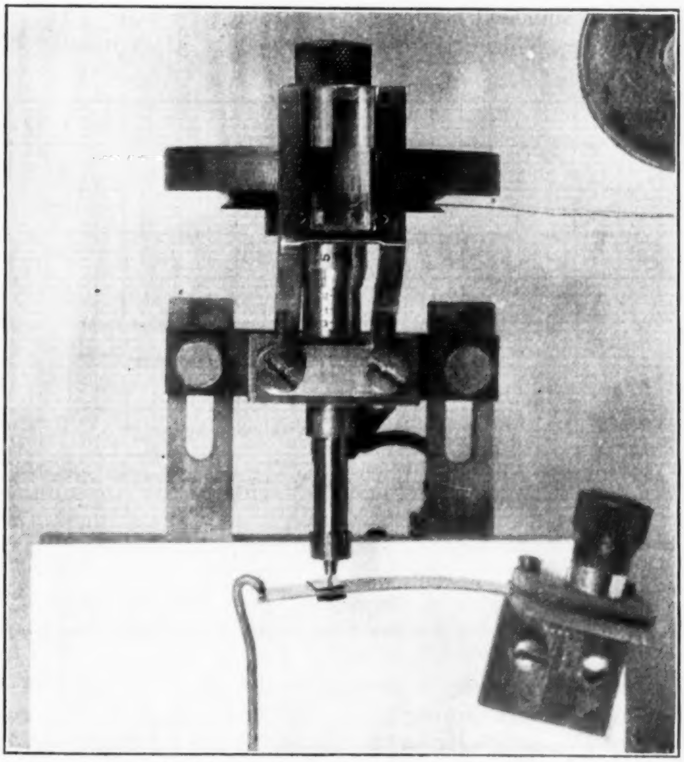

FIG. 3.—The Usual Method of Supporting a Flat-Strip Specimen so that Displacements Are Essentially Vertical and Parallel to the Movement of the Micrometer Spindle.

DIFFERENCE BETWEEN SENSITIVITY AND ACCURACY

It is recognized that this measuring method does not record deflections accurately to a millionth of an inch from some zero load position. This would require far greater accuracy than is obtainable in a micrometer screw thread. However, the percentage of error is small, and it is not accuracy but sensitivity that is important. For example, if the total hysteresis measured in a test is 100 millionths or 0.0001 in., a millionth of an inch sensitivity is 1 per cent of the total, which is sufficient accuracy for drift measurement. This distinction between accuracy and sensitivity is important, and of course limits the usefulness of the method to a relatively small range of displacement.

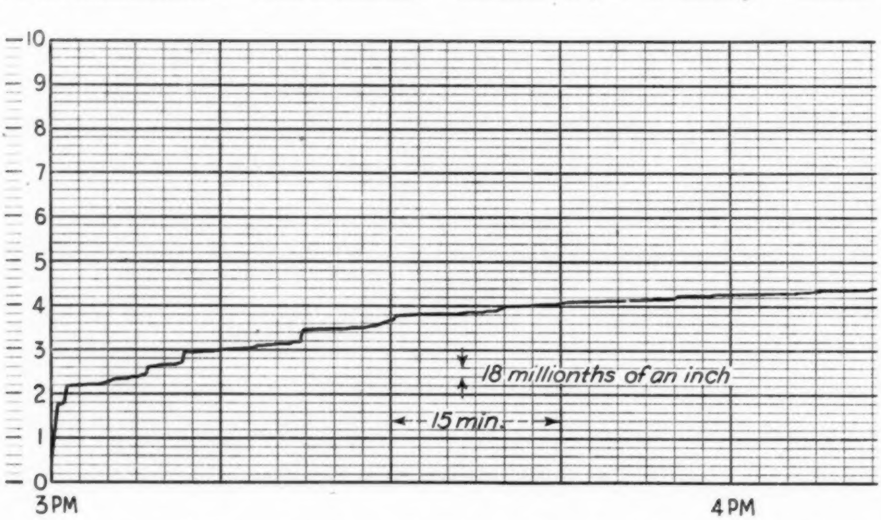

FIG. 4.—Typical Chart Record of Elastic Drift in a Cantilever Beam Specimen.
Breaks in the line represent actual deviations of the specimen, and are typical of all materials.

RESULTS OF TYPICAL TESTS

Measurements of elastic drift have been made with the electronic micrometer on a wide variety of non-ferrous spring materials as well as on many types of complete instrument elements. Tests on spring materials are made on specimens loaded as cantilever beams, as shown in Fig. 3. The load is applied and the micrometer engaged quickly to record the initial changes in deflection. Then the test is continued automatically or the period desired, giving a chart record such as the portion of one shown in Fig. 4, for a non-ferrous alloy strip. This chart shows the fundamental characteristic of drift as it is found in all spring materials under usual stress conditions: the rate of drift decreases with increasing time under load. For greater convenience, it is usually desirable to scale the chart and plot the curve to a condensed time scale, as has been done in Fig. 5 for a typical test on a heat-treated beryllium copper specimen. In this curve, drift is shown in percentage of load deflection rather than the measured displacement since this is a more useful basis for comparing the performance of different specimens.

The curve in Fig. 5 flattens out, indicating that drift might stop at some extended time. However, by plotting the same curve to a logarithmic time axis, as shown in Fig. 6, the points fall on a straight line. Experience with tests continued without interruption for as long as 15 days shows that if measurements are made with sufficient sensitivity, drift never ceases, although it may in time decrease to such a slow rate that it has no further significance. If the initial points on the curve are determined accurately, the curve may be projected to indicate the amount of drift at the end of a much longer period of time.

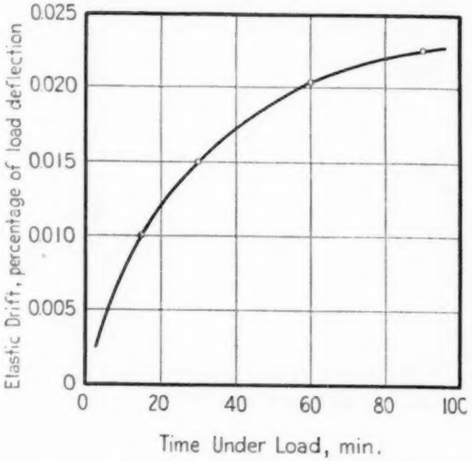

FIG. 5.—Drift in a Heat-Treated Beryllium-Copper Strip.

Expressed in percentage of load deflection to simplify comparison with specimens of different dimensions and other materials.

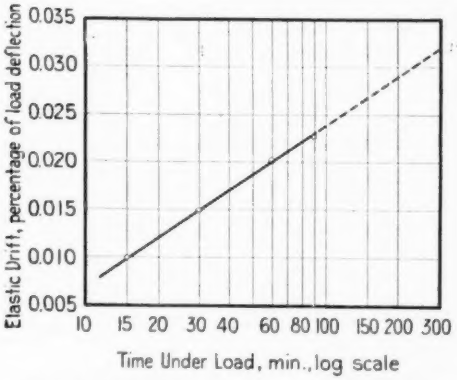

FIG. 6.—The Curve Shown in Fig. 5 Plotted to Log of Time Becomes a Straight Line, Indicating that Drift Continues as Long as the Load Remains.

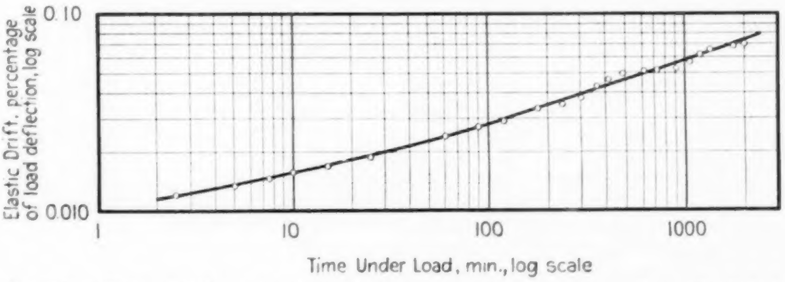

FIG. 7.—Long-Time Drift Test of a Metal Bellows Plotted from Data Scaled from the Chart Record.

Variations in plotted points are caused by small changes in specimen temperature.

For more involved stress conditions such as found in flexible metal bellows for example, continuing increase in drift under load may take the form of the curve shown in Fig. 7, where logarithmic scales are used for both the time and deflection. Waves in this curve are the result of small changes in specimen temperature between day and night conditions, and of course can be eliminated if desired by thermostatic control of temperature.

From the manner in which these curves are plotted, it would appear that there was a known initial or drift-free deflection point from which hysteresis measurements were made. However, drift proceeds so rapidly immediately after applying the load that the recording mechanism cannot keep up with the changes. Therefore, the actual recorded deflection at some time after applying the load, usually from 15 sec. to 2 min., is taken as the starting point. It would appear desirable to determine the rate at which drift takes place during the first few seconds after applying the load, but a satisfactory method has not yet been developed using the electronic micrometer.

During the first few minutes of a test, particularly where the amount of drift is large, the recorded curve usually shows breaks in the steady rate of deviation, such as shown in Fig. 4. Curves usually average out to a smooth continuous line, but the deviations appear to hold back and then let go. After a test has proceeded for 10 or 15 min., such breaks are seldom found. External disturbances sometimes cause operation of the recording mechanism, but these disturbances are readily recognized, and do not affect the final shape of the curve.

Investigation of the factors affecting drift shows that internal stress conditions are by far the most important, particularly in cold-worked materials. The effect of heat treatment, time under load, stress intensity, type of loading and repeated cycles of load, as well as the characteristic drift in the various materials have been studied. Routine tests have been made to check performance of production spring elements. Production tests on the deflection rate of various types of springs have been made with extreme precision. Since the measuring apparatus places no load on the spring, it can be used as effectively on small, weak springs as on large ones.

Acknowledgment.—Acknowledgment is made to L. L. Stott of the Beryllium Corporation of Pennsylvania for permission to reproduce the curves shown in Figs. 5 and 6.

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R. W. Carson, "Better Instrument Springs," *Transactions*, Am. Inst. Electrical Engrs., Vol. 52, p. 869 (1933).

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L. C. Tyte, "Extension of Metal Wires Under Longitudinal Stress," *Philosophical Magazine*, Series 7, Vol. 17, p. 634 (1934).

Stress-strain ratio is exponential. Deviations attributed to plastic distortion in unfavorably situated grains.

M. F. Sayre, "Laws of Elastic Behavior," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 56, p. 559 (1934).

Supplies a rational explanation of hysteresis and drift based on molecular conditions within grains and on disturbed conditions at grain boundaries.

DISCUSSION

Mr. A. B. Smith[1] (*presented in written form*).—Mr. Carson is to be commended for his treatment of definitions at the very beginning of his paper. Included in this matter is his definition of "rapidly" connected with the separation between "elastic hysteresis" and "elastic drift." The former is independent of time only if the time is short enough—which he later defines as "10 to 30 min." (see p. 665).

I note that Mr. Carson finds that the drift curve seems to be logarithmic. If so, it cannot agree with experimental data at zero time. It is logical to believe that the deflection due to elastic drift is zero at zero time, whereas the logarithmic function starts at negative infinity (since the base is usually greater than unity).

Though the above is not reasonable, I have had a similar experience in magnetic measurements, in which the mathematical expression which most nearly fitted the experimental curve had negative values at zero time. It was not illogical, because of the well-known shape of such curves in iron in that region of time. Does the true elastic drift curve have a similar shape?

It would be interesting to know the magnitude of the opposing forces at the point where the deflections were measured, as well as the total deflection from the zero of the spring.

The use of the international units of weights and measures would have greatly improved the presentation of this valuable contribution.

Mr. M. F. Sayre.[2]—I should like first to express my admiration for the way in which the author has handled this rather difficult question of automatic measurement of extremely small quantities. Then I should like to challenge, in some measure, the title of the article in which the author speaks of "measuring elastic drift." I have been measuring these low-temperature changes for some time past and comparing them with high-temperature changes, and I confess I do not yet see wherein there is any sufficient physical difference in their character to justify the introduction of a new and separate name. I have a feeling that unless creep is to be specifically defined as a "slow change in shape resulting from recrystallization under stress," this is a creep just as truly as high-temperature measurements are creep. A large part of this creep is recoverable on removal of the load, but I think that our high-temperature experimenters are just beginning to realize that a very large percentage of the high-temperature creep is also recoverable on removal of the load.

A second word in the title should also probably be changed. I think that this should be spoken of as pseudo-elastic rather than elastic drift. It is a change in dimension which very gradually occurs, which represents some form either of slip on crystal planes within the crystals or of gradual change in molecular position, which is of a different type from elastic changes. On removal of the load, the induced forces

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[2] Professor of Applied Mechanics, Union College, Schenectady, N. Y.

produced by the original movement cause a reverse movement. The percentage of return movement is largely a question of the previous history of the material. For a material in an absolutely virgin state the percentage would probably be appreciably less than is shown here. On the other hand, I should defy anyone to manufacture a specimen in the shape needed for a test and have it in the virgin condition. Simply the process of handling is likely to produce stresses such as would modify the condition. What is being measured here is the change in length, in some measure, as influenced by previous history. The residual creep or drift under load, however, is frequently a very important property of the material, and Mr. Carson's method has very much facilitated its measurement.

MR. L. B. TUCKERMAN.[3]—First I wish to commend the author upon adding a very convenient method to some of the less convenient methods which have been used in the past for measuring the inelastic phenomena which occur in metals under relatively low stresses. The importance of more convenient methods of measuring these inelastic effects from the standpoint of the production of accurate measuring instruments, cannot be over-estimated. Anyone who makes this type of measurement easier is making it possible to study the phenomena more thoroughly and improve the accuracy of instruments.

Just as an illustration of the problems which persons are up against when they try to make accurate instruments, take the case of a sensitive aneroid barograph used as an altimeter. At the National Bureau of Standards we are placed in the position of having to take the barograph records from record flights and say approximately how high an airplane went. Because of elastic drift, we cannot do this by comparing the flight record with a previous calibration of the instrument. The only way that we can get a sufficiently accurate result is to put the instrument in a chamber and reproduce the same record by carrying it again through the same sequence of temperatures and pressures, measuring the pressures on accurate barometers of the mercury type. In other words, the drift—and I prefer the word drift to creep—the drift of those instruments depends so much upon the rate of loading, the time rate of change of temperature and all the detailed conditions of the application of the load to the spring, that it is necessary to repeat the same sequence if you wish to know accurately what the readings mean. Now it is true that aneroid barographs are greatly improved over what they were twenty years ago, because people have been studying these phenomena, and I welcome the thought of a spring which can be loaded. What was the stretch of that spring that drifted only three-millionths of an inch?

MR. ROBERT W. CARSON.[4]—That was loaded at about 50 lb. per sq. in. in torsion.

MR. TUCKERMAN.—Well, that's going some. If we can study these things, and study them conveniently, we probably will have more springs with these improved characteristics, and, perhaps, might even hope some time to have an aneroid barograph which we could bring down to ground and take the record at face value.

I do not agree with Mr. Sayre that we should call this phenomenon "creep." In my mind the word creep is associated with slow inelastic yielding which does not recover on unloading. In that con-

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[4] Instrument Specialities Co., Little Falls, N. J.

nection I rather question whether, if the drift were observed for an infinite time, a curve such as Fig. 6 would continue to be a straight line. An infinite time is a long time, but some of the records of drift I have seen show that even with a logarithmic time scale the plot definitely curves downward, approaching a horizontal line. We have held some materials under constant load for as much as seven months, and although after seven months the drift was still continuing it was slowing down so much that we anticipated that perhaps after a hundred years it would be reasonably negligible. I doubt whether it continues under these very low loads quite so fast—I mean logarithmically fast—as would be indicated by the extrapolation of the curves that he shows here.

MR. P. G. MCVETTY.[5]—Mr. Carson's work refers particularly to a drift in instrument springs. Attempts to correlate this with the creep of metals under stress at high temperatures is made difficult by our lack of knowledge of the exact nature of creep. Mr. R. W. Bailey has defined creep as "the outward manifestation of the balance in the destruction of strain hardening by thermal influence and its recreation by further slip." According to this definition, the two phenomena may be correlated if there is enough softening effect at room temperature to remove part of the strain hardening in the spring. Whether or not this is true it appears that the author has developed a good method of measuring the deflections of instrument springs.

Another point has to do with the plotting of results. If I understood the author correctly, he recommends the logarithmic plotting of Fig. 7 to convert the curve of Fig. 5 into a straight line. Reference to Mr. Boyd's discussion of Mr. Marin's[6] paper shows that the logarithmic plot introduces a kink in the curve if the time be prolonged. This should be considered before adopting the logarithmic plot.

The use of this apparatus over long periods of time raises questions as to the effect of dirt or corrosion at the contact points. There is a possibility also of relative movement between the support for the micrometer and the support for the cantilever beam specimen. Such details could be worked out easily if long-time use of the equipment were desired.

A MEMBER.—I wonder whether the author has applied this test method, with the contacts through his electrical circuit, to plastics which ordinarily would not have electrical conductivity. Is there some way of maintaining an active contact between the micrometer point and the electric circuit, in order to measure such materials as plastics that do not have electrical conductivity to any degree?

MR. CARSON (*author's closure*).—Differences between hysteresis and drift or creep can be readily defined in terms of the observed effect, to answer Mr. Smith's question. Hysteresis is for all practical purposes instantaneous and is readily observed as the difference between deflections for ascending and descending loads of the same magnitude. Drift or creep is most readily observed as a continuing slow increase in deflection under a steady load, and starts immediately upon applying the load. The logarithmic form of the drift or creep curve cannot ordinarily be projected back much closer than about two minutes after applying the load. Drift or creep during this initial period ap-

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[6] See p. 265.

pears to follow some other law, merging gradually into the logarithmic function as time increases. Electronic micrometer tests indicate that the creep during this initial period proceeds at a slower rate than obtained by projecting the logarithmic function back to zero time.

The author agrees with Mr. Sayre that both of the words "elastic" and "drift" in the title of the paper are poorly selected. They should be replaced with the single word "creep", which should be defined to include both the continuing increase in deflection under a constant load, as well as the recovery effect found upon removal of the load, whether the specimen is tested at normal temperatures or at some elevated temperature.

Many investigators of high temperature creep have assumed, as has Mr. Tuckerman, that there is no reversal of creep when the load is removed or reduced. The author suggested several years ago[7] that this assumption was incorrect, and recent investigations of the relaxation problem have shown that creep reversal does take place in high-temperature tests as well as in the normal temperature loading described in this paper.

It is understandable that one familiar with high-temperature creep effects in metals would define the effect in terms of the factors that happened to be important in his special problem. The definition of creep quoted by Mr. McVetty is certainly applicable to high-temperature tests on steel, but cannot be used as a general definition of the term "creep". It is the author's experience that all engineering materials will exhibit creep under any load at any temperature—if the measuring equipment is sufficiently sensitive to find the creep.

With the present knowledge on "inelastic" properties of materials, creep must now be defined to include effects measured under normal temperatures as well as at high temperatures, and for other materials than steel. As a general definition the author proposes: Creep is a continuing change in the deflection of a stressed member under a constant load.

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[7] *Mechanical Engineering*, June, 1934, p. 365; *Metals and Alloys*, July, 1934, p. 374.

A CLASSIFICATION OF METHODS OF MECHANICAL ANALYSIS OF PARTICULATE MATERIALS[1]

By Paul S. Roller[2]

The physical state of a particulate substance related to the quality known as fineness is characterized by the three physical constants of surface area per unit weight, number of particles per unit weight, and uniformity of the size distribution. For these constants to have their proper utility in interpreting and controlling technical processes, it is necessary that they be accurately stated. To this end it is necessary that the size distribution data on which they are based be precise, and above all free of systematic errors of method. The recent finding of a law of size distribution **(1)**[3] by which it is possible from a knowledge of two or more experimental points to determine true integral values of the above stated physical constants makes it particularly desirable that the experimental data be unequivocal.

Many methods of mechanical analysis have been proposed and used. Some of these are much more capable of giving a correct result than others. It is believed that a classification of methods of size analysis offers the best way of determining the reliability and respective merits of different methods. In the classification proposed, the methods are divided into different classes, depending on the underlying principles involved. Each class is discussed from the standpoint of adequacy of principle and of technique.

References have been cited to give a recent representative example of each of the methods; no attempt has been made at complete citations nor is it implied that the example given represents necessarily the best technique for a given method. Previous references are usually given in those cited. A valuable bibliography has been compiled by Krumbein **(2)**.

Classification of Methods of Size Analysis

A concise statement of the different classes of measurement and of the principles underlying each class may be made as follows:

Class I.—Direct Observation by Means of the Microscope. No principles involved other than those pertaining to the use of the microscope.

Class II.—Fractionation and Recovery of the Fractions. The only principle involved is that the velocity of fall varies with size of particle, the relationship being expressed quantitatively by Stokes' law.

Class III.—Sedimentation with Direct Physical Measurement of the Rate of Settling. This method of measurement depends also only on Stokes' law, but there is no separation into fractions as for Class II. The conditions of measurement and interpretation are also different from those that apply to Class II.

Class IV.—Sedimentation with Indirect Measurement of the Rate of Settling. This class is similar to Class III,

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[3] The boldface numbers in parentheses refer to the reports and papers given in the list of references appended to this paper, see p. 681.

except that in addition to Stokes' law, other principles are brought in to determine the result.

Class V.—Measurement of Surface Area of the Material, Without Determining the Size Distribution. The principle on which the measurement is based is physico-chemical.

DISCUSSION OF THE METHODS OF CLASS I

By measuring with the microscope the size of each of an assembly of particles a size distribution is obtained and from this, as mentioned above, the physical constants of surface area, number of particles, and uniformity may be derived. In consideration of these properties it is clearly necessary that the size of a given particle be correctly defined, and indeed in such a way that the definition bears some simple relationship to the physical property of interest.

In the case of a sphere or of a cube the diameter or the edge is the obvious magnitude to measure, and also the correct one since it is related simply to the surface or volume of the particle. In the case of irregular particles, the three dimensions,—length, width and depth—should be measured. Knowing the three dimensions, a quantity may be derived that is related to the volume or surface area of the particle. Unless the three dimensions are actually measured and suitably averaged, a quite incorrect value for the size of the particle may be obtained. For example, the author has observed cases (3) in which one of the dimensions of a particle is as little as one-third of the mean size based on surface and one of the other dimensions as great as fifteen times the mean. In such cases, from the standpoint of a true result only a suitable mean obtained from all three dimensions can be considered.

Requirements in measuring the diameter of a sphere, and methods of measuring the depth of an irregular particle, are discussed elsewhere (3).

All techniques of microscopic measurement have in common the necessity of properly dispersing the sample on a slide, or in a counting chamber. Otherwise the size of agglomerates rather than of discrete particles will be measured. For dispersion, any suitable chemical dispersing agent may be used. This must be accompanied by mechanical agitation, in which operation care must be taken that the small grains are not crushed.

With heterogeneously distributed material it is necessary to change from one objective to another, and to recalculate the results to a uniform basis (4). To avoid this insecure procedure, the expedient is frequently adopted of first fractionating the material according to the methods of Class II. Naturally only a crude fractionation is considered necessary, but it is obvious that there are limits of crudity beyond which compensation cannot be secured by the exacting use of the microscope, for example as regards loss of material during fractionation.

In measurement with the microscope, three main methods and one secondary method may be distinguished: (*A*) direct observation, (*B*) projection on a screen, (*C*) photomicrography, and (*a*) counting in a counting chamber.

Method A.—A ruled ocular net may be used for estimating the size of the grain (4).

Method B.—This method is considerably more convenient than method *A*. The particle is measured with a portable rule or with a net ruled on the screen (5). Each particle is conveniently brought into separate focus by remote control.

Method C.—This method (6) is evidently suitable only for highly uniform materials or at least for materials in which the variation in depth is less than the depth of focus of the system. The depth of focus operative in photomicrography is given by $\frac{1}{8MA}$ where M is the total magnification and A is the numerical aperture (7) and is small relative to the size of particle. The depth of the particles cannot of course be measured by this method.

Method a.—Since a size distribution is not obtained in this method (8) it is regarded as subordinate to methods A, B and C. On non-uniform material an average size is calculated that is in the main determined by the more numerous small particles. This average is hardly related to the average size that is obtained in considering the dimensions of the individual particles and which for non-uniform distributions is the most significant.

Discussion of Methods of Class II

In methods of Class II, the material is separated into size fractions by an upward stream of fluid, assuming Stokes' law of fall. Means for deflocculation must be present. The flow is continued until complete separation is obtained. The fractions are weighed and may be examined under the microscope whenever a check is desired of the actual sizes against the theoretical. Such a check should not be necessary in a tested system. For different particle shapes, the relationship between the Stokes' law size which is obtained in a fractionation and the size of particle related to volume or surface area per unit volume has been studied elsewhere (3); in general the relationship may be deduced by microscopic examination of a closely sized fraction. For cubic particles the mean edge is approximately the common measure of size.

The methods to be distinguished in this class of measurement are: (A) fractionation with air, (B) fractionation with liquid, usually water, and (C) sedimentation and decantation. Temperature control is needed in methods B and C but is relatively unimportant in method A.

Method A.—Deflocculation is obtained by means of the energy of the air jet. This energy must not be so great as to disrupt the individual crystals, a matter which is controlled by regulating the pressure. The energy of the jet is sufficient to move the sample of powder out of the field of action, hence provision must be made to automatically return the sample into the path of the jet and into the field of action (9). Adherence of dust is satisfactorily avoided by the use of stainless steel polished tubes, electrical grounding, and vibration (9).

Method B.—In order effectively to liberate the particles, use of a jet of fluid and a deflocculating agent and mechanical agitation are required (10). In the presence of such agitation, a possible particle break-down and the effects of turbulence must be tested for. The rate of separation is intrinsically less than in method A, since the rate of fall of a particle in water is roughly one-hundredth of that in air.

Method C.—Sedimentation and Decantation. At calculated intervals a part of the suspension is siphoned or flowed off, thus recovering part of the particles below a given size. The flow of liquid may be considered discontinuous instead of continuous as for the preceding methods. Many siphonings are necessary for any given size, the number being determined to a large extent by the efficiency of deflocculation. This makes the method time consuming. The appreciable time of siphoning introduces an unavoidable error which cannot be mitigated by increasing the rate of siphoning, since in so doing eddy currents and capture of settled particles result (11).

DISCUSSION OF THE METHODS OF CLASS III

Class III methods of measurement are distinguished from those of Class II in two main respects. In the first place, the concentration of particles at a given level rather than discrete separation is used to measure the percentage of material having a given particle size. In the second place deflocculation must be made complete in one step, and must remain so during sedimentation. In the methods of Class II a longer time of fractionation compensates for imperfect deflocculation. This dissimilar relation to deflocculation between Classes II and III has not been given the recognition it merits.

It is well known that different deflocculating agents have different degrees of effectiveness (13), while a given deflocculating agent naturally will act more or less differently with different materials, with changes in condition of the surface of a given material, with change in mechanical aids to deflocculation, and with change in the number concentration of the particles in suspension (12). Criteria should therefore be established to determine under what conditions one-step deflocculation as practiced in the methods of Class III is really complete and remains so.

An increase in the efficiency of deflocculation should be reflected in an increase in the turbidity of a suspension, increase in the electro-kinetic potential (negative cataphoretic velocity) of the particles, and in a decrease in the sedimentation volume. These conditions may be used as guides in determining the extent of deflocculation. Probably the most satisfactory test is to compare results with one of the more positive methods of Classes I and II. Microscopic examination of a sample for this class of measurement is not a satisfactory test of flocculation as it yields no information as to the percentage of fine material that has flocculated and settled past the level of measurement.

The methods of Class III may be divided according to the means that are used to measure the concentration of the suspension at a given level and time. Temperature control is necessary in all cases. The methods may be divided as follows: (*A*) pipette, (*B*) balance, (*C*) hydrometer, (*D*) manometer.

Method A.—This method seems to have been independently devised by different investigators at about the same time (2), but received a high form of development in the apparatus of Andreasen (13, 14). The pipette is used to withdraw samples at a given level. The solid is separated from the liquid by evaporation and is then weighed. This method is considered of the highest rank because of the relative simplicity of measuring the concentration and of calculating the size distribution.

Method B.—The settled particles are weighed in a balance pan (15, 16), a curve of weight against time constructed, and the size distribution determined by drawing tangents to the curve and taking the difference of the slopes. The drawing of tangents is made necessary by the fact that this method gives a record of the whole sedimenting column instead of conditions at a given level, as in method *A*. It has been shown that due to concentration currents, some of the material may settle around the pan instead of on it (17, 18).

Method C.—The density of the suspension at a given level is measured by means of a hydrometer. As in method *A*, the drawing of tangents is not necessary, since the density measured is that at a given level. Because of the latter condition it is imperative that the dimensions of the bulb be small (19) compared to those of the vessel. For this reason the large bulbed Bouyoucos hydrometer (20) which was originally intended for the special purpose of determining "soil colloids" is distinctly not applicable. The settling of particles on the hydrometer bulb requires that consideration be given to the shape of the bulb (21). Disturbance caused by removal of the hydrometer should also be considered.

Method D.—The density of the suspension is measured by means of a side-arm manometer (22). Tangents must be drawn as in method *B*. Large displacements of the manometer fluid may be secured by use of an inclined tube (23) or by the use of two liquids (24), but the resistance due to viscosity and surface tension and consequent lag of the manometer readings must be contended with (25). By measuring the density between two neighboring points (26), the necessity of drawing tangents is avoided, but the relative displacements are small, and the method is therefore unusually sensitive to small variations.

DISCUSSION OF THE METHODS OF CLASS IV

The methods of Class IV are similar in principle to those of Class III, except that the concentration of the suspension at a given level is measured by means other than those of the direct physical ones of Class III. These means, in fact, are based on special properties of the material in suspension. In this way new principles are introduced into the procedure which are additional to those of Stokes' law of fall. Because these additional principles inevitably complicate and throw suspicion on the final result, this class of measurement is ranked below that of Class III.

As an example of the considerations that enter into measurements of this class, we may consider the method based on measurement of light absorption. In this method the diameter is determined by Stokes' law of fall while measurement of the concentration is made to depend on the principle that the light absorption is inversely proportional to the surface reflection of the material. Actually, however, the final result depends on surface reflection, surface absorption, volume transmission, light scattering or diffraction, and the effects of multiple reflection. The assumption that these factors are constant for different sizes and concentrations is not beyond doubt.

Methods that come under this class are:

Method A.—Light Absorption. The density of the suspension at a given level or levels is determined by the degree of absorption of the light (27). An obvious variant of this method is to measure the light scattered at right angles to the incoming beam.

Method B.—Light Depolarization. The ratio of intensities of the two polarized components of the Tyndall beam is measured (28). Shape of particle and concentration affect the results.

Method C.—Dielectric Constant. The dielectric constant of the suspension at a given level is measured, and from this is derived the volume concentration of material at that level (29). Adsorption appears to affect the measured dielectric constant (30).

DISCUSSION OF THE METHODS OF CLASS V

In this class the surface area of the particulate material is measured by reference to some property, such as adsorption, that is proportional to the surface area of the powder. Because a size distribution is not obtained, this class of measurement is ranked last. However, this does not mean that it is the least important, as it has individual applications of its own.

It should be noted that the surface area measured by this class of methods is not necessarily the same as the area measured by the preceding classes; in fact generally is not the same. In Classes I to IV the area derived from the measurements is a geometric one; in Class V the area that is measured is determined by all the accessible surface and thus includes the sub-microscopic and molecular crevices and irregularities. Therefore the latter area will as a rule be greater than the former. If one is desirous of obtaining a value for the "surface roughness" of a material, a comparison of the surface measured by the methods of Class V and the geometric surface as measured by a method of one of the other classes would be useful.

For all of the methods of Class V a reference must be used from which the observed result is translated into surface area per gram. This reference may consist of some general principle such as that adsorption takes place in a unimolecular layer, or it may consist of the use of a standard-size fraction of known surface such as may be obtained by one of the methods of Class II. In the present state of our knowledge the latter procedure is preferable. Effects due to change in chemical nature or contamination of the surface or to impurities in general should be guarded against.

A statement of the methods of this class may be given as follows:

Method A.—Adsorption of Dyes. The amount adsorbed is conveniently determined by the methods of colorimetry (**31, 32**).

Method B.—Adsorption of Heterohomopolar Molecules. An example is the adsorption of oleic acid from solution and determination of the amount adsorbed by acid-base titration (**33**).

Method C.—Adsorption of Vapors and Gases (**34**).

Method D.—Exchange Adsorption of Metal Powders with Metal Atoms. The surface atom of a metallic powder may exchange with nobler ions in solution, the latter apparently forming a monomolecular layer on the metal surface (**35**).

Method E.—Rate of Solution (**36**). It should be noted that the measured rate may be affected by the structure of the dissolving sediment (**37**).

Method F.—Heat of Wetting by a Liquid (**38**).

Method G.—Radiochemical Methods. Exchange Adsorption (**39**). The adsorption of radium ion by isotopic lead compounds, and possibly also by compounds isomorphous with these, is a measure of the surface of the material. Emanating Power (**40**): The compound whose surface is to be measured is prepared with an addition of radium salt and the emanating power of the compound so prepared is measured. The method appears applicable to isotropic and amorphous compounds as well as to isotopic lead compounds (**41**).

Method H.—Measurement of the Coercive Force in the Case of Magnetic Substances (**42**).

CONSIDERATIONS IN SELECTING A METHOD OF SIZE ANALYSIS

The selection of a method of analysis will depend on what extent completeness of data, precision, absolute correctness, and speed of operation are considered desirable. Speed of operation, in fact, cannot be considered independently of completeness and accuracy, for a sacrifice in the latter two factors is prompted by a desire to gain speed.

For a quick semi-quantitative result, examination of the material may be made under the microscope or better compared under a comparison microscope with a material of known fineness.

An accurate but incomplete estimate of the fineness is had by sieving through a No. 200 sieve. One may also measure the content of material below some very small size. In this way, one determines the "concentration" of fines, a quantity which is opposed to the "residue" of coarse obtained in an ordinary sieve analysis. The concentration of fines should usually be considered more important than the residue of coarse because it is the significant quantity in determining the physical properties of surface area and number of particles per gram. For a great many materials a limiting size of 5μ may be conveniently used as reference.

Determination of the concentration of fines should find its greatest application in operating practice where a quick index of milling operation is desired. One of the methods of Classes II, III and IV would seem most convenient for this determination; the methods of Class IV should, however, be employed with due regard to the reservation next discussed.

In the determination of complete size distribution, the aim of obtaining a true result demands that the methods be generally limited to those of Classes I, II and III. The methods of Class IV should be considered only after the accessory principles of this class are proved to be quite invariant. The use of the microscope appears in general to be most suitable for materials of fairly uniform distribution. Certain of the methods of Class III have been used to determine size distribution down to about 0.1μ, while the lower limit recorded for the methods of Class II is 2μ. Therefore the methods of Class III appear to be the more suitable for extremely fine materials. However, in so far as the methods of Class III are more troubled by problems of deflocculation, they are at a disadvantage relative to the methods of Class II.

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SUMMARY OF PROCEEDINGS OF SYMPOSIUM ON CONSISTENCY: CRITICAL DISCUSSION ON PRESENT-DAY PRACTICES IN CONSISTENCY MEASUREMENT

The Symposium on Consistency was held at the second session of the Fortieth Annual Meeting of the American Society for Testing Materials at New York City, on June 29, 1937. The excellent attendance throughout the symposium session was a definite indication of the widespread interest in this subject.

The symposium was sponsored by Technical Committee II on Consistency, Plasticity and Related Properties, of Committee E-1 on Methods of Testing. Detailed arrangements for the symposium papers were made by E. C. Bingham, Chairman of Technical Committee II, and W. H. Fulweiler, Chairman of Committee E-1.

Mr. Fulweiler served as chairman of the symposium session and was assisted by the following co-chairmen: E. C. Bingham, H. A. Gardner, C. S. Reeve, L. C. Beard, Arthur W. Carpenter, and T. Smith Taylor.

In introducing the Symposium on Consistency, Mr. Fulweiler summarized briefly the reasons for arranging for this series of papers to consider the advances in recent years in consistency measurement, including a discussion of the theoretical background and the existing nomenclature. He called attention to the fact that the nine papers comprising the symposium covered a representative cross-section of engineering materials concerning which the establishment of authoritative nomenclature and fundamental principles underlying the measurement of consistency, plasticity, viscosity and related properties is important. Mr. Fulweiler stated that it was the purpose of the symposium to present a critical review of consistency measurement as applied to those materials, and to consider the possibilities of standardization and the use and advantage to be derived from the use of fundamental units of measurement.

Mr. Fulweiler then introduced the first paper, following which the remaining papers were presided over in turn by the co-chairmen. The papers comprising the symposium were as follows:

"Recent Progress in Consistency Measurement," by E. C. Bingham, Professor of Chemistry, Lafayette College.

"Definition of Consistency and Theoretical Considerations," by M. Mooney, Development Dept., U. S. Rubber Products, Inc.

"Consistency Measurements in the Paint Industry," by D. L. Gamble, Research Division, New Jersey Zinc Co.

"The Flow Properties of Asphalts Measured in Absolute Units," by R. N. Traxler, Research Division, Technical Bureau, The Barber Co.

"Consistency Measurements in the Coal-Tar Industry," by E. O. Rhodes, E. W. Volkmann and C. T. Barker, Technical Dept., Tar and Chemical Division, Koppers Co.

"Viscosity Measurement of Petroleum Products and Lubricants," by J. C. Geniesse, Research Chemist, The Atlantic Refining Co.

"Consistency Measurement of Rubber

and Rubber Compounds," by J. H. Dillon and L. V. Cooper, Firestone Tire and Rubber Co.

"Measurements of Flow Characteristics of Thermosetting Resins," by H. L. Bender, H. F. Wakefield and H. E. Riley, Bakelite Corp.

"Cold Flow of Insulating Materials," by Robert Burns and Irving L. Hopkins, members of the Technical Staff, Bell Telephone Laboratories, Inc.

Mr. Bingham in reviewing briefly the progress in consistency measurement that has taken place since the first Symposium on Consistency[1] presented before the Society in 1923, emphasized the need for a standard of viscosity. He pointed out that water at 20 C. has been suggested but that there is lack of agreement as to what the correct value should be, resulting in an uncertainty of nearly 0.5 per cent. A difference of this magnitude is intolerable since viscosimeters are being developed in which viscosity measurements are reported to the fifth significant figure. It is therefore imperative in such work to state what value is taken as standard. He pointed out the advantages of the simple pipette type of instrument which depends upon the hydrostatic head for the flow of the liquid, and advocated the use of two- or three-point calibration for capillary viscosimeters.

Mr. Mooney discussed the subject from a scientific or theoretical point of view. He pointed out that the flow properties of a material can only be scientifically expressed in the form of its rheological diagram, or some equivalent, which shows the variation in rate of shear with shearing stress in a continuous simple shearing deformation. Since "viscosity" applies to the simple liquids only, he stated that for the more complex materials the term "consistency" is needed. He defined consistency as the ratio of shearing stress to the rate of shear. The term is thus similar to "viscosity," but is used when the ratio referred to varies with the shearing stress, while "viscosity" is used only when the ratio is constant.

Mr. Mooney presented an analysis of the various rheological tests established by the Society for materials having complex rheological diagrams, such as, plastic solids, plastico-viscous solids, and complex liquids. Of the 26 recognized methods summarized it is evident that for the most part they are not of the "scientific" type which measures some specific physical property of the material nor of the "practical" type which attempts to parallel closely some service condition, but they are rather of a "hybrid" type. In the author's opinion there is little that can be said to justify the "hybrid" type of test since they will neither predict with certainty the behavior of the material in complex service conditions, nor permit reliable calculations in simple conditions that can be analyzed.

The author recommends that any test should either be thoroughly practical or truly scientific. It is believed that measurements can be made in fundamental or absolute units for this as well as for other physical properties under normal conditions. The apparatus required may or may not be more complicated than that already in use, but even if more complicated, it will by measuring fundamental properties quantitatively, give more complete and more reliable information than has been available heretofore.

Mr. Gamble discussed consistency measurements in the paint industry, where the conditions vary all of the way from those in enamels to those met in flat-wall paints. The former have so little internal structure that a single

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part II, p. 432 (1923).

measurement of the apparent viscosity is often sufficient for plant control. The latter has higher pigmentation, a flocculated character and often other structural materials, such as a soap gel, hence the apparent viscosity (that is, the consistency) changes rapidly with the shearing stress. Many of the instruments used by the paint industry are not closely related to the scientific or absolute type. An instrument which gives results in absolute units is advocated.

Mr. Traxler, in discussing the flow properties of asphalts, pointed out that the asphalt technologist is confronted with the problem of measuring a wide range of consistencies. He discusses methods which have been used successfully for measuring in absolute units, low and high viscosities and for evaluating the flow of non-viscous materials. The capillary tube or rotating cylinder type of viscosimeter is recommended for low viscosities (less than 50,000 poises). The latter instrument or a viscosimeter utilizing the principle of the falling coaxial cylinder is suggested for measuring the viscosity of highly viscous asphalts. Non-viscous bitumens (for example, most air-blown asphalts) can best be evaluated using a rotating cylinder type of viscosimeter.

A suitable plastometer must separate not only the yield value and mobility as independent variables but also eliminate the elasticity and adhesiveness. It is possible to separate those materials which possess a definite yield value from those which behave like simple viscous liquids. Both thixotropy and elasticity (the non-permanent deformation) can be followed and measured. The change of consistency with the temperature (susceptibility) and the evaluation of mineral fillers in mixtures afford problems which have been successfully attacked by means of absolute instruments, of which the coaxial cylinder type of instrument has proved most useful.

Messrs. Rhodes, Volkmann and Barker discussed the consistency testing methods used by the coal-tar industry which are empirical and include such methods as the Engler specific viscosity test, float test, softening point by ring-and-ball, and cube-in-air or cube-in-water test, penetration test, and Saybolt Furol viscosity test. Correlation between these tests is difficult because of the empirical character of the results obtained. The authors point out that a rational evaluation of viscosity testing is possible only by the use of absolute units, and that reliable information as to the conversion of results obtained into absolute or fundamental units is needed. It is shown that the softening point temperatures (ring-and-ball) cannot be related accurately to absolute viscosities, and that satisfactory conversion of penetration measurements is problematical. The relation between float test seconds and kinematic viscosity, however, is sufficiently well defined to be of practical interest. Engler degrees and Saybolt seconds can be converted into absolute units. The authors presented a chart developed for the conversion of Saybolt Universal and Saybolt Furol viscosity values into kinematic viscosity, which shows the conversion relations for Redwood No. 1 and British Road Tar Association viscosimeter seconds. Experimental evidence is presented to show that the viscosity-temperature relations of coal tars and coal-tar products can be plotted as straight lines on this chart. Since tars appear to be of the nature of viscous liquids, questions as to the yield value and rate of shear are of little concern up to 108 poises. The simplicity of the viscosity-temperature relation also emphasizes the need for determination of the viscosity of coal tars.

The paper by Mr. Geniesse deals with viscometry practices in the petroleum industry. He pointed out that while the Saybolt Universal and Saybolt Furol viscosimeters are extensively used in the industry and have been standardized by the Society, they have a number of shortcomings and inherent difficulties which cannot be entirely eliminated. The Saybolt instrument is inaccurate at high temperatures because the lower end of the outflow tube is exposed to the atmosphere, and also at short times of outflow due to the high kinetic energy correction. The fact that the oil volume is measured after it has cooled considerably below the temperature of the test introduces another complication, making it difficult to compare the results of the Saybolt apparatus with any other instrument. A third complication results from the fact that the Saybolt viscosity of an oil is the time of outflow, under prescribed conditions, from a metal tube whose dimensions are specified but which must be duplicated to a very high degree of accuracy in order to be checked in another similar instrument. Other instruments of the capillary tube type which measure kinematic viscosity in centistokes are being introduced and should eliminate many of the errors unavoidable in the older methods. Since the petroleum industry will continue for some time the use of the "Saybolt seconds" it is necessary to have conversion tables and charts which have accordingly been developed. The charts have a range in kinematic viscosity of from 0.4 to 20,000,000 centistokes, and in temperature from −30 F. to 450 F.

In discussing the use of the pentrometer for testing greases, the author pointed out the real need for knowledge of their flow under rates of shear encountered in service. A simple instrument giving results in absolute units would reduce the number of scales required and would be very welcome. A consideration of the problems still facing the rheologist leads the author to the conclusion that the most important one is the adoption of an international scale of viscosity units. It would eliminate considerable expensive laboratory testing and simplify the preparation of specifications. The next most important need is for a commercial viscosimeter to measure consistency at various controlled rates of shear for plastic liquids and solids.

The last three papers of the symposium deal with the rheological measurements in the more difficult field of the plastic materials, and discuss tests for rubber and rubber products, the thermosetting resins, and insulating materials.

Messrs. Dillon and Cooper confine their discussion to that of the consistency of crude and unvulcanized rubber compounds. The problems discussed concern "break down" on shear, the thixotropic increase in consistency of the rubber on standing, slippage, and prevulcanization. Since rubber is plastic, the measurement of the consistency is complicated, but nevertheless the measurement in absolute units promises to yield needed information in regard to the structure of rubber and to give a better basis for comparison. The authors discuss several semi-absolute plastometers as well as some others that are quite empirical. For example, in the Mooney plastometer they find that there is thixotropic equilibrium and slippage is taken care of. With further improvement, the shearing cylinder and disk instrument might prove satisfactory for absolute measurement. There is pressing need for instruments to correlate with factory conditions, but even with satisfactory instruments for control, the logical procedure should be to concentrate on the development of a satisfactory research instrument. Once that instrument is developed, the problem of adapting it to control requirements could be attacked.

The paper by Messrs. Bender, Wakefield and Riley reviews the status of rheology in the field of the thermosetting resins. The stable resins may be melted, dissolved or flowed out without great differences in the flow conditions, so that the measurement of the consistency in absolute units offers no great difficulty. The heat-stable resins show thixotropy and the structure is altered by solution. They are plastic, so that measurement of the consistency is less easy. But greater difficulties of measurement are offered by the resins which are not heat stable due to polymerization, secondary growth of structure, and an increase in mobility due to "self-orientation" which opposes the first two effects which both tend to decrease the mobility. These resinous plastics are all thermoplastic but many of them are also thermosetting or chemo-plastic in that they lose their property of plasticity at elevated temperatures. Therefore, at a given temperature and shearing stress, a given plastic increases in mobility, passes through a maximum and decreases rapidly to zero, as a result of the opposing tendencies. It is necessary to measure these changes while in progress at working temperatures to understand what is taking place. The authors, therefore describe numerous tests and present a résumé of the various methods used for determination of the arbitrary flow values, for expressing the summation of the flow value, change of flow value, and change of hardening speed with time.

Messrs. Burns and Hopkins discussed the slow flow which takes place at nominal shearing stresses in materials used as insulators in the electrical industry. On telephone switch-boards, for example, several electrical contacts may have to be broken in rapid and reproducible sequence over a period of 15 yr. or more, where the total movement is only 0.02 to 0.03 in. This "cold flow" is therefore important. It is necessary to distinguish between elasticity, viscosity, elastic recovery and permanent set and the material must show a cold flow below a certain specified amount. From detailed studies of cold flow phenomena, the authors conclude that methods of parallel plate plastometry are of fundamental importance in determining the class in which a given material falls, but are too laborious and complicated for engineering or production control. The test described in the paper, in which the 24-hr. deformation is taken, although an empirical one, has proved an accurate and reliable method of evaluating insulating materials subject to deformation under heat and pressure.

The papers, complete with discussion have been published by the Society as a separate volume entitled, "Symposium on Consistency: Discussion on Present-Day Practices in Consistency Measurement."

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